ΥΠΟΜΕΤΡΟ 16.1 – 16.2 "ΊΔΡΥΣΗ ΚΑΙ ΛΕΙΤΟΥΡΓΙΑ ΕΠΙΧΕΙΡΗΣΙΑΚΩΝ ΟΜΑΔΩΝ ΤΗΣ ΕΥΡΩΠΑΪΚΗΣ ΣΥΜΠΡΑΞΗΣ ΚΑΙΝΟΤΟΜΙΑΣ ΓΙΑ ΤΗΝ ΠΑΡΑΓΩΓΙΚΟΤΗΤΑ ΚΑΙ ΤΗ ΒΙΩΣΙΜΟΤΗΤΑ ΤΗΣ ΓΕΩΡΓΙΑΣ"

ΔΡΑΣΗ 2: «Υλοποίηση του επιχειρησιακού σχεδίου (project) των Επιχειρησιακών Ομάδων της ΕΣΚ για την παραγωγικότητα και βιωσιμότητα της γεωργίας»

Επιχειρησιακή Ομάδα: InnoDry

Καινοτόμα μετασυλλεκτική εφαρμογή καταπόνησης χαμηλής θερμοκρασίας για τον φυσικό εμπλουτισμό προϊόντων καρυδιάς (*Juglans regia* L.)

ΕΛΓΟ-ΔΗΜΗΤΡΑ
ΕΛΛΗΝΙΚΟΣ ΓΕΩΡΓΙΚΟΣ ΟΡΓΑΝΙΣΜΟΣ - ΔΗΜΗΤΡΑ
ΙΤΑΠ

Συντάκτες: Μ. Χριστόπουλος, Γ. Ουζουνίδου

Παραδοτέο 1.1: Εκπαιδευτικό υλικό λυοφιλίωσης

Έκδοση: 1 Μάρτιος 2023

ΕΥΡΩΠΑΪΚΟ ΓΕΩΡΓΙΚΟ ΤΑΜΕΙΟ ΑΓΡΟΤΙΚΗΣ ΑΝΑΠΤΥΞΗΣ
Η Ευρώπη επενδύει στις αγροτικές περιοχές

ΕΛΛΗΝΙΚΗ ΔΗΜΟΚΡΑΤΙΑ
ΥΠΟΥΡΓΕΙΟ ΑΓΡΟΤΙΚΗΣ ΑΝΑΠΤΥΞΗΣ ΚΑΙ ΤΡΟΦΙΜΩΝ

eip-agri

ΕΣΠΑ 2014-2020
ανάπτυξη - εργασία - αλληλεγγύη

1. Εισαγωγή

- Η ξήρανση (αφυδάτωση) είναι μια αρχαία διαδικασία που χρησιμοποιείται για τη συντήρηση των τροφίμων.
- Με την ξήρανση παράγονται προϊόντα με μειωμένη περιεκτικότητα σε υγρασία (νερό) και ανάλογα με την νωπή-μη αποξηραμένη πρώτη ύλη (είδος και χαρακτηριστικά αυτής) και το επιδιωκόμενο τελικό προϊόν (μορφή, υφή, εμφάνιση, τρόπος διάθεσης κλπ.) προσδιορίζονται τα επιθυμητά τελικά επίπεδα υγρασίας ώστε τα τελικά αφυδατωμένα να έχουν παρατεταμένη διάρκεια ζωής.
- Για να γίνει απομάκρυνση του νερού απαιτείται ενέργεια η οποία παρέχεται εξωγενώς στην πρώτη ύλη.
- Η συμβατική μέθοδος ξήρανσης πρώτης ύλης φυτικής προέλευσης είναι η θερμική με την οποία η απαιτούμενη ενέργεια για την απομάκρυνση του νερού από το προϊόν παρέχεται μέσω θέρμανσης της πρώτης ύλης.
- Η πιο απλή μέθοδος θερμικής αποξήρανσης που χρησιμοποιείται για χιλιάδες χρόνια είναι με έκθεση της πρώτης ύλης στον ήλιο (π.χ. σταφίδες, ξηροί καρποί κλπ.)
- Ανάλογα με τίς συνθήκες (θερμοκρασία, ατμόσφαιρα, έκθεση στο φως κλπ.) που γίνεται η ξήρανση οδηγεί σε μεταβολές της πρώτης ύλης οι οποίες μπορεί να είναι αντιληπτές ή μη αντιληπτές από τον καταναλωτή του τελικού αποξηραμένου προϊόντος.
- Αντιληπτές από τον καταναλωτή μεταβολές μπορεί να αφορούν την εμφάνιση (π.χ. συρρίκνωση, αλλαγή χρώματος κλπ.)
- Μη αντιληπτές από τον καταναλωτή μεταβολές αφορούν αλλαγή στην ποσότητα ή/και το είδος των συστατικών που περιέχει το τελικό αποξηραμένο προϊόν σε σχέση τη νωπή πρώτη ύλη (π.χ. βιταμίνες, αντιοξειδωτικά συστατικά κλπ.)
- Οι μη αντιληπτές από τον καταναλωτή μεταβολές μπορεί να υποβαθμίζουν άμεσα τη θρεπτική αξία (μη αντιληπτό χαρακτηριστικό ποιότητας) του προϊόντος (π.χ. η ξήρανση υπό συγκεκριμένες συνθήκες οδηγεί σε ‘καταστροφή’ βιταμινών)
- Οι μη αντιληπτές από τον καταναλωτή μεταβολές μπορεί να υποβαθμίζουν έμμεσα και την αντιληπτή από τον καταναλωτή ποιότητα (π.χ. η ξήρανση υπό συγκεκριμένες συνθήκες οδηγεί σε οξείδωση φαινολικών συστατικών όπου τα συστατικά που προκύπτουν μαυρίζουν το προϊόν)

2. Παράγοντες που επηρεάζουν την ποιότητα ενός αποξηραμένου προϊόντος

❑ Η τελική ποιότητα ενός αποξηραμένου προϊόντος επηρεάζεται από:

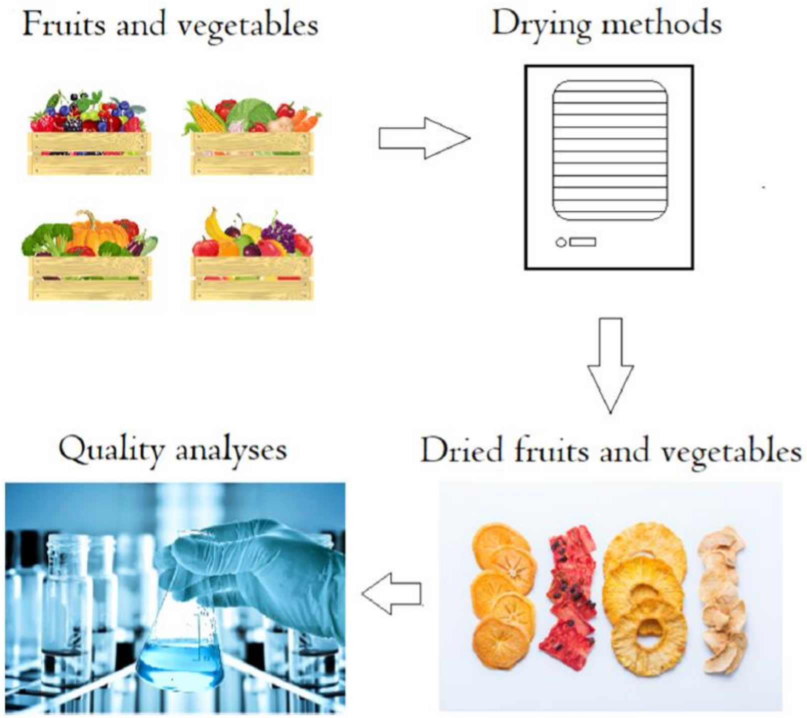
1. Ποιότητα αρχικής πρώτης ύλης (π.χ. μορφή, εμφάνιση, περιεκτικότητα σε νερό, σύσταση κλπ.)
2. Διαδικασίες-Συνθήκες κατά την αποξήρανση
3. Διαδικασίες-Συνθήκες μετά την αποξήρανση μέχρι την κατανάλωση

❑ Οι βασικοί παράγοντες κατά την αποξήρανση που επηρεάζουν την ποιότητα ενός αποξηραμένου προϊόντος:

1. Τρόπος και ταχύτητα απομάκρυνσης του νερού από τη νωπή πρώτη ύλη
2. Θερμοκρασία αποξήρανσης
3. Σύσταση αέρα στο χώρο που γίνεται η αποξήρανση (κυρίως το ποσοστό O2)
4. ‘Υπαρξη και ένταση φωτός στο χώρο που γίνεται η αποξήρανση (κυρίως το ποσοστό O2)
5. Δράση άλλων φυσικών μεγεθών (π.χ. πίεση, ακτινοβολία κλπ.)

❑ Οι παραπάνω βασικοί παράγοντες μπορεί να δρουν ανεξάρτητα ο ένας από τον άλλο ή και συνεργιστικά (π.χ. η αύξηση των επιπέδων ενός παράγοντα να μειώνει ή να αυξάνει τη δράση άλλου (ων) παραγόντων.

❑ Η ανάπτυξη της επιστήμης και της τεχνολογίας έχει δώσει τις δυνατότητες οι παραπάνω παράγοντες να μπορούν να ελέγχονται πλήρως ή μερικώς κατά τη διαδικασία αποξήρανσης.

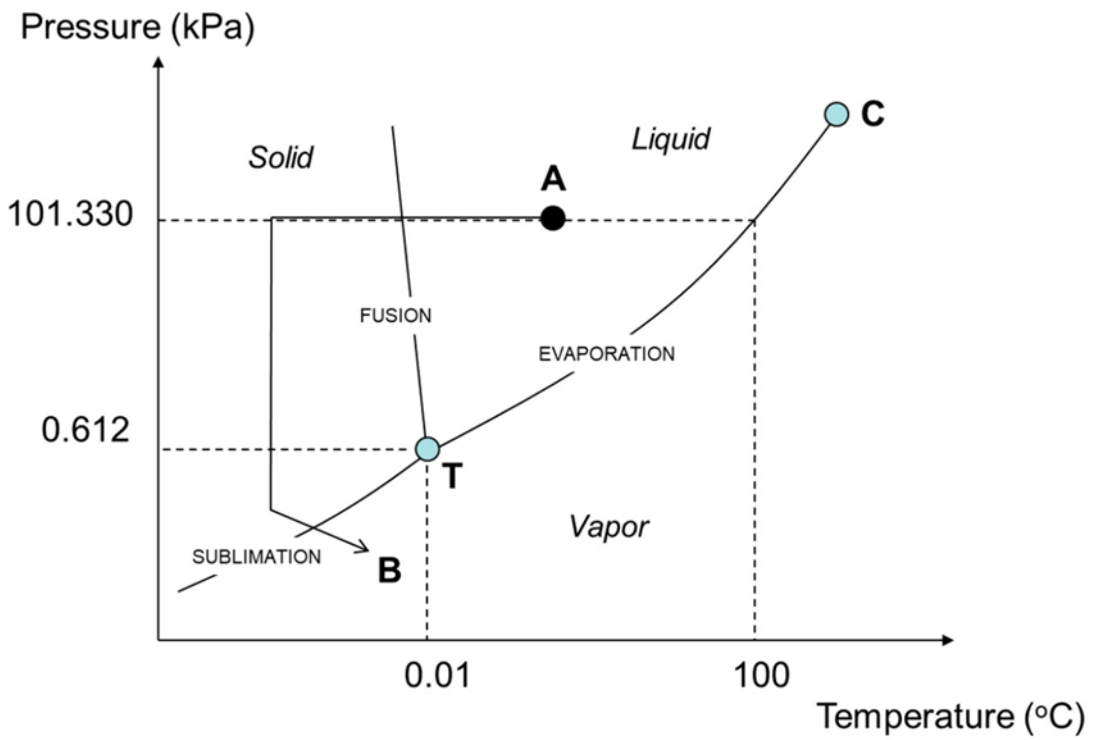


Πηγή: Richter Reis, F., Marques, C., Moraes, A.C.S.d., Masson, M.L., 2022. Trends in quality assessment and drying methods used for fruits and vegetables. Food Control 142, 109254/

- Η λυοφιλίωση είναι μια από τις νέες τεχνολογίες αποξήρανσης
- Τα τελευταία χρόνια έχει αρχίσει και αναπτύσσεται δυναμικά η χρήση της λυοφιλίωσης και στον τομέα της αποξήρανσης φυτικών προϊόντων
- Η λυοφιλίωση βασίζεται στην αφυδάτωση μέσω εξάχνωσης ενός κατεψυγμένου προϊόντος
- Λόγω της απουσίας νερού στην υγρή μορφή και των χαμηλών θερμοκρασιών που απαιτούνται για τη διαδικασία, διακόπτονται οι περισσότερες χημικές-βιοχημικές και μικροβιολογικές επιδράσεις με αποτέλεσμα να προκύπτει ένα τελικό προϊόν εξαιρετικής ποιότητας

Πηγή: https://www.ourpaleolife.com/freeze-dried-vs-dehydrated/

- Το νερό υπάρχει σε τρεις διαφορετικές καταστάσεις: στερεό, υγρό ή αέριο (ατμός).
- Το σχήμα 1 παρουσιάζει το διάγραμμα φάσης του νερού (πίεση έναντι θερμοκρασίας), όπου οι γραμμές καμπύλης δείχνουν το πέρασμα από στερεό σε ατμό (εξάχνωση), από υγρό σε ατμό (εξάτμιση) ή από στερεό σε υγρό (σύντηξη).
- Το σημείο T στο σχήμα 1 αντιπροσωπεύει το τριπλό σημείο του νερού (στους 0,01 °C και 0,612 kPa) όπου συνυπάρχουν οι τρεις φάσεις (υγρό, ατμός, στερεό) και το σημείο C είναι το κρίσιμο σημείο του νερού (374 °C και 22060 kPa).
- Η λυοφιλίωση χρησιμοποιεί το φαινόμενο της εξάχνωσης (σε θερμοκρασίες χαμηλότερες από 0,01 °C και πιέσεις υδρατμών κάτω από 0,612 kPa).
- Στο σχήμα 1, ένα προϊόν που πρόκειται να λυοφιλιωθεί θα ακολουθήσει τη διαδρομή από το A στο σημείο B (δηλαδή, το προϊόν θα πρέπει πρώτα να καταψυχθεί μειώνοντας τη θερμοκρασία του και μετά η πίεση των υδρατμών θα πρέπει να μειωθεί κάτω από την πίεση που αντιστοιχεί στο τριπλό σημείο και τέλος θα πρέπει να παρέχεται κάποια θερμότητα για να βοηθήσει τον πάγο να μετατραπεί σε ατμό με εξάχνωση).



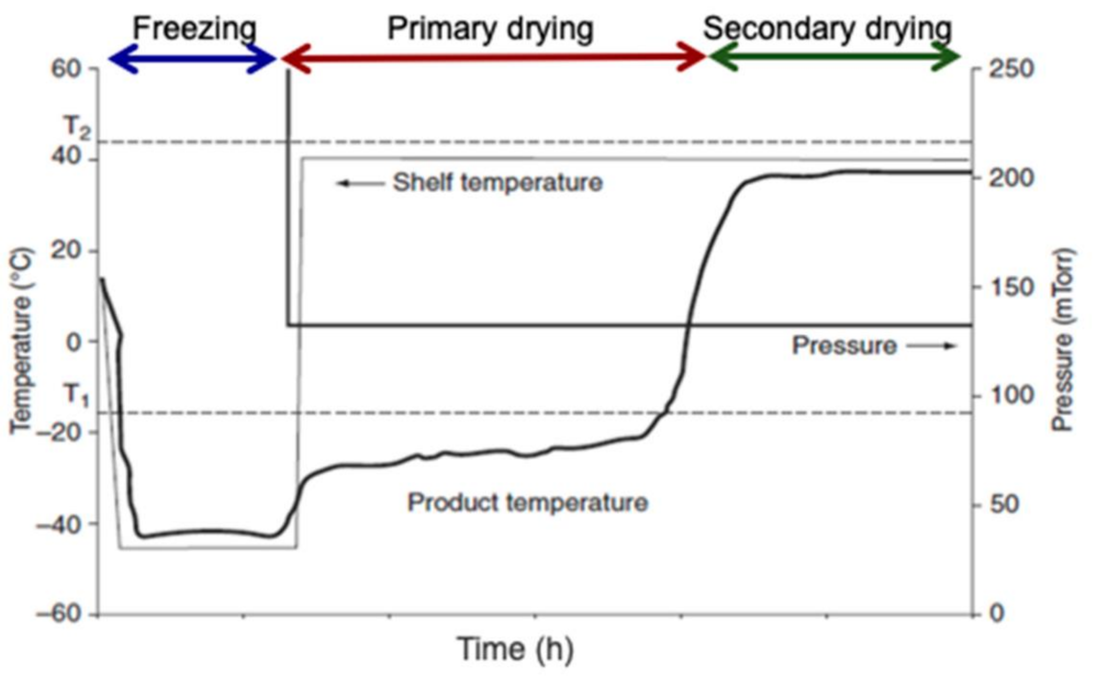
Σχήμα 1. Διάγραμμα φάσεων νερού (T: τριπλό σημείο νερού, C: κρίσιμο σημείο νερού). Το "A" αντιπροσωπεύει το σημείο εκκίνησης πριν από την λυοφιλίωση (ατμοσφαιρική πίεση και θερμοκρασία περιβάλλοντος), ενώ το "B", τις επιθυμητές τελικές συνθήκες κατά την εξάχνωση (κάτω από το τριπλό σημείο T) [Από: (Bhatta et al., 2020)].

- Κατά τη διαδικασία λυοφιλίωσης, η απομάκρυνση του νερού στερεάς κατάστασης (πάγος) πραγματοποιείται σε τρία στάδια:

(α) κατάψυξη, όπου το δείγμα πρέπει να καταψυχθεί πλήρως.

(β) πρωτογενής (κυρίως) ξήρανση, όταν ο πάγος εξαχνώνεται, συνήθως σε υποατμοσφαιρική πίεση

(γ) δευτερεύουσα (τελική) ξήρανση, όταν το υπόλοιπο μη παγωμένο/δεσμευμένο νερό εκροφάται από το ξηρότερο πλέγμα (matrix) τροφίμων



Σχήμα 2. Στάδια λυοφιλίωσης [Από: (Bhatta et al., 2020)]

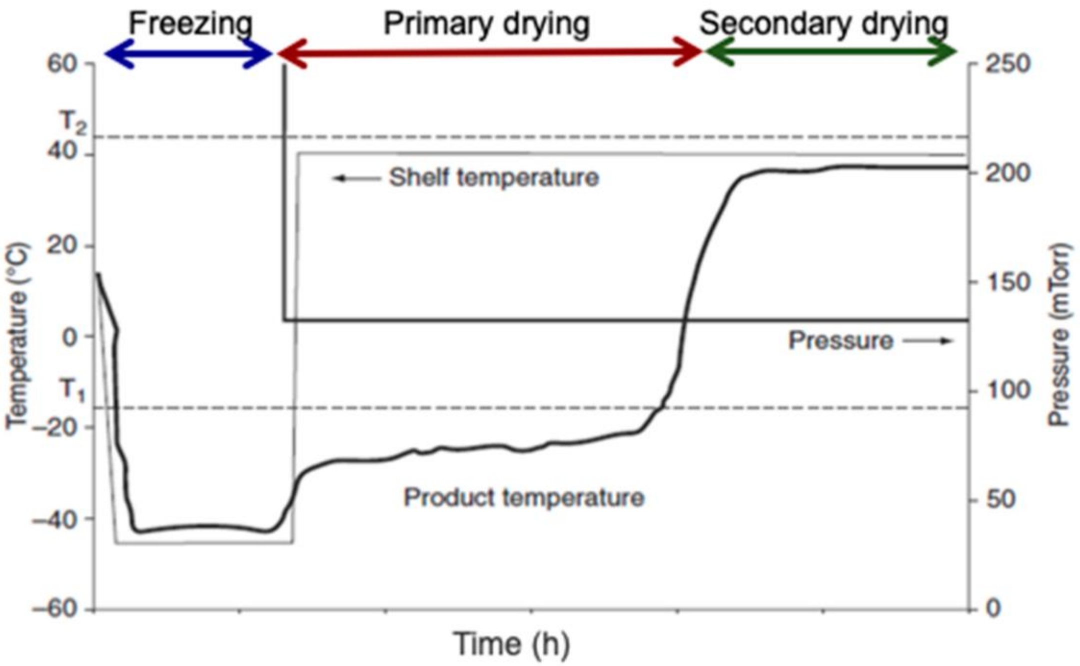
4. Θεωρητικό υπόβαθρο λυοφυλίωσης (στάδιο κατάψυξης)

- Η κατάψυξη είναι το πρώτο βήμα διαχωρισμού στη διαδικασία λυοφιλίωσης, η οποία στερεοποιεί τα συστατικά των ιστών.

- Ο ρυθμός κατάψυξης είναι σημαντικός για το σχηματισμό και το μέγεθος των κρυστάλλων πάγου

- Με αργό ρυθμό κατάψυξης σχηματίζονται μεγαλύτεροι κρύσταλλοι πάγου και αντίστροφα.

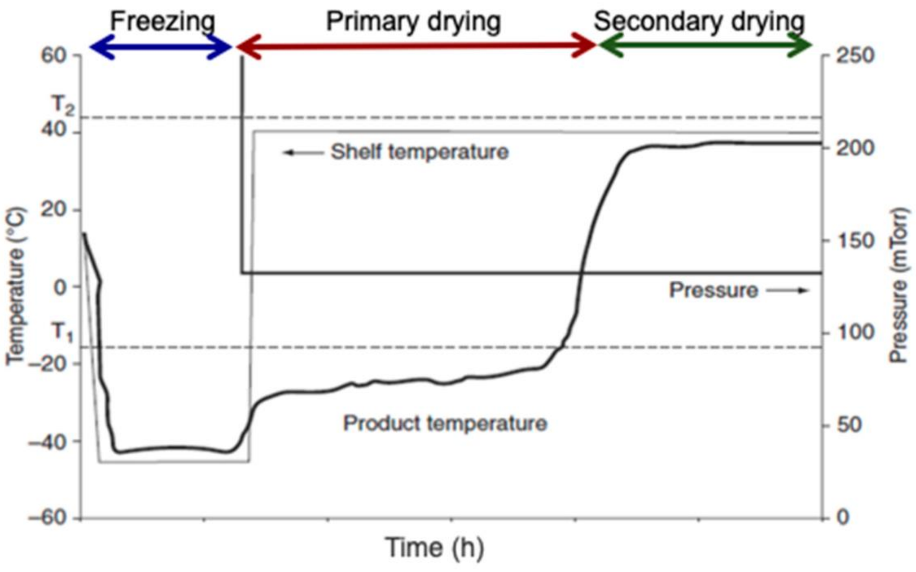
- Κατά συνέπεια, το μέγεθος των κρυστάλλων επηρεάζει τον ρυθμό ξήρανσης, όπου οι μεγάλοι κρύσταλλοι πάγου είναι ευκολότερο να εξαχνωθούν και ως εκ τούτου αυξάνουν τον ρυθμό της πρωτογενούς ξήρανσης (Bhandari et al., 2013).



Σχήμα 2. Προφίλ θερμοκρασίας του προϊόντος κατά τη διάρκεια της διαδικασίας λυοφιλίωσης, όπου T1 (διακεκομμένη γραμμή) είναι η θερμοκρασία κατάρρευσης και T2 (διακεκομμένη γραμμή) είναι η θερμοκρασία υαλώδους μετάπτωσης των ξηρών στερεών [Από: (Bhatta et al., 2020)]

4. Θεωρητικό υπόβαθρο λυοφυλίωσης (στάδιο κυρίως ξήρανσης)

- Στην κυρίως ξήρανση, εφαρμόζεται ένα κενό και η θερμοκρασία του ραφιού αυξάνεται για να ξεκινήσει η εξάχνωση, έτσι ώστε η θερμοκρασία του προϊόντος να είναι 2–3 °C κάτω από τη θερμοκρασία κατάρρευσης Tc (Patel et al., 2010, Tang and Pikal, 2004).
- Η θερμοκρασία κατάρρευσης είναι η θερμοκρασία πάνω από την οποία το προϊόν έχει τον κίνδυνο να χάσει τη μακροσκοπική του δομή κατά τη διαδικασία λυοφιλίωσης.
- Η Tc θα μπορούσε να προσδιοριστεί με ένα μικροσκόπιο λυοφιλίωσης, αλλά μπορεί επίσης να εκτιμηθεί από τη θερμοκρασία υαλώδους μετάπτωσης (Tg).
- Θα πρέπει να σημειωθεί ότι η Tc θα μπορούσε να είναι 2 °C έως 20 °C υψηλότερη από την Tg, ανάλογα κυρίως με τη σύσταση του δείγματος (Roos and Drusch, 2015).
- Ωστόσο, πολύ συντηρητικές προβλέψεις της Tc μπορεί να οδηγήσουν σε πολύ μεγαλύτερη χρονικά διαδικασία λυοφιλίωσης, επομένως μπορεί να χρησιμοποιηθεί μόνο σε κρίσιμες περιπτώσεις όταν το δείγμα είναι δύσκολο λυοφιλιωθεί.
- Το Σχήμα 2 απεικονίζει το τυπικό προφίλ θερμοκρασίας ενός προϊόντος κατά τη διάρκεια κάθε σταδίου της διαδικασίας λυοφιλίωσης, όπου μπορεί να παρατηρηθεί ότι κατά την αρχική ξήρανση η θερμοκρασία του προϊόντος πρέπει να είναι κάτω από τη θερμοκρασία κατάρρευσης (που αναπαρίσταται ως διακεκομμένη γραμμή T1 στο Σχήμα 2).



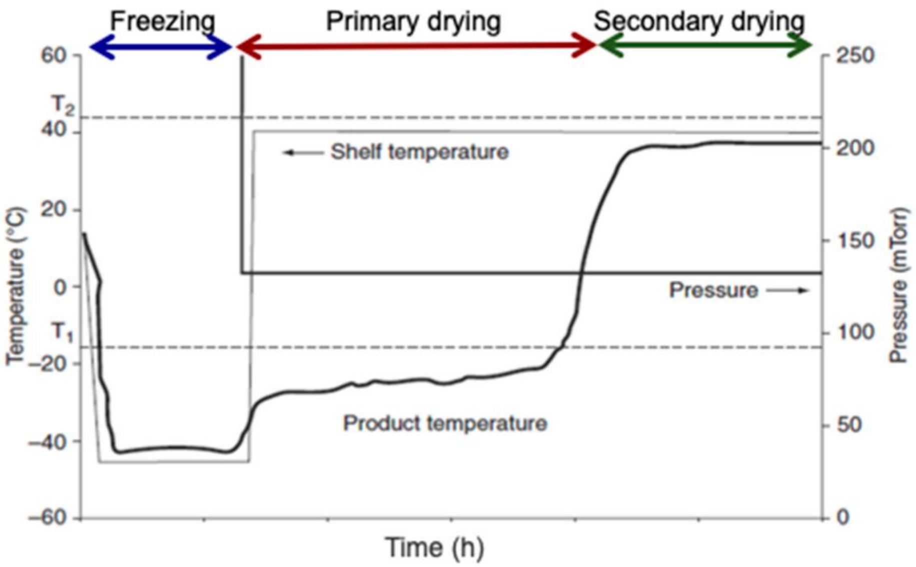
Σχήμα 2. Προφίλ θερμοκρασίας του προϊόντος κατά τη διάρκεια της διαδικασίας λυοφιλίωσης, όπου T1 (διακεκομμένη γραμμή) είναι η θερμοκρασία κατάρρευσης και T2 (διακεκομμένη γραμμή) είναι η θερμοκρασία υαλώδους μετάπτωσης των ξηρών στερεών [Από: (Bhatta et al., 2020)]

4. Θεωρητικό υπόβαθρο λυοφυλίωσης (στάδιο τελικής ξήρανσης)

- Η τελική ξήρανση ξεκινά όταν η εξάχνωση λαμβάνει ακόμα χώρα, καθώς αποτελεί αργό μέρος της διαδικασίας λυοφιλίωσης, η οποία μπορεί να χρειαστεί τουλάχιστον 30% περισσότερο χρόνο για να ολοκληρωθεί από το τέλος της εξάχνωσης.

- Αυτό το τελευταίο βήμα θα μπορούσε να εκτελεστεί σε αυξημένη θερμοκρασία ραφιού για να αφαιρεθεί πιο αποτελεσματικά το υπόλοιπο μη παγωμένο ή δεσμευμένο νερό με εκρόφηση, αλλά χαμηλότερη από τη θερμοκρασία υαλώδους μετάπτωσης των ξηρών στερεών (που παρουσιάζεται ως διακεκομμένη γραμμή T2 στο Σχήμα 2).

- Ωστόσο, είναι δύσκολο να προσδιοριστεί το τελικό σημείο της πρωτογενούς ξήρανσης ή η έναρξη της δευτερογενούς φάσης ξήρανσης. Εάν η θερμοκρασία αυξηθεί πριν εξαχνωθεί όλος ο πάγος (τελικό σημείο της αρχικής φάσης ξήρανσης), θα μπορούσε να οδηγήσει σε κατάρρευση του προϊόντος και υποβάθμισης της ποιότητας του.



Σχήμα 2. Προφίλ θερμοκρασίας του προϊόντος κατά τη διάρκεια της διαδικασίας λυοφιλίωσης, όπου T1 (διακεκομμένη γραμμή) είναι η θερμοκρασία κατάρρευσης και T2 (διακεκομμένη γραμμή) είναι η θερμοκρασία υαλώδους μετάπτωσης των ξηρών στερεών [Από: (Bhatta et al., 2020)]

6. Γενική μεθοδολογία υλοποίησης σχετικά με την λυοφιλίωση στα πλαίσια του ΕΣ

- ΕΕ1 Εκπαίδευση-μεταφορά τεχνογνωσίας

Π1.1 Εκπαιδευτικό υλικό λυοφιλίωσης

Π1.3 Εκπαίδευση παραγωγικών φορέων στη μέθοδο της λυοφιλίωσης (θεωρητική και πρακτική)

- ΕΕ2 Σχεδιασμός, βελτιστοποίηση, ex situ παραγωγής αποξηραμένων προϊόντων με τη μέθοδο λυοφιλίωσης

Π2.1 Σχέδιο πρωτοκόλλου λυοφιλίωσης

Π2.2 Τελικό πρωτόκολλο λυοφιλίωσης

- ΕΕ4 Σχεδιασμός, βελτιστοποίηση, in situ παραγωγή αποξηραμένων προϊόντων με τη μέθοδο λυοφιλίωσης

Π4.1 Έκθεση 1ης πιλοτικής in situ παραγωγής αποξηραμένων προϊόντων με τη μέθοδο λυοφιλίωσης

Π4.2 Έκθεση 2ης πιλοτικής in situ παραγωγής αποξηραμένων προϊόντων με τη μέθοδο λυοφιλίωσης

- ΕΕ6 Αξιολόγηση παραγωγικής διαδικασίας και τελικών προϊόντων των δύο πιλοτικά εφαρμοζόμενων καινοτόμων μεθόδων αποξήρανσης

Π6.1 Αξιολόγηση παραγωγικής διαδικασίας και τελικών προϊόντων λυοφιλίωσης

Π6.3 Μελέτη σταθερότητας τελικών αποξηραμένων προϊόντων

7. Προτεινόμενη βιβλιογραφία για περαιτέρω θεωρητική εμβάθυνση

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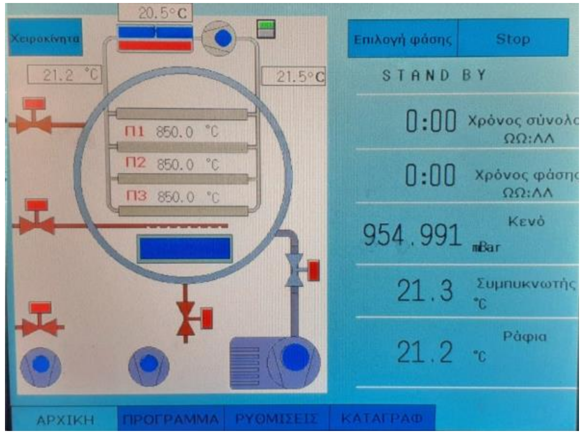
7. Φωτογραφικό παράρτημα

Συσκευή λυοφιλίωσης εργαστηριακής κλίμακας

Συσκευή λυοφιλίωσης παραγωγικής κλίμακας

Συσκευή λυοφιλίωσης πιλοτικής κλίμακας

- Στα πλαίσια υλοποίησης του ΕΣ InnoDry θα αξιοποιηθούν συσκευές λυοφιλίωσης εργαστηριακής και πιλοτικής κλίμακας



Πάνελ χειρισμού και ελέγχου συνθηκών λυοφιλίωσης

7. Φωτογραφικό παράρτημα

Εργαστηριακής κλίμακας λυοφιλίωση

Πιλοτικής κλίμακας λυοφιλίωση

Τελικά αποξηραμένα καρύδια με τη μέθοδο της λυοφιλίωσης (αριστερά) και της συμβατικής θερμικής αποξήρανσης (δεξιά)

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REVIEW ARTICLE

Recent advances in drying and dehydration of fruits and vegetables: a review

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Abstract Fruits and vegetables are dried to enhance storage stability, minimize packaging requirement and reduce transport weight. Preservation of fruits and vegetables through drying based on sun and solar drying techniques which cause poor quality and product contamination. Energy consumption and quality of dried products are critical parameters in the selection of drying process. An optimum drying system for the preparation of quality dehydrated products is cost effective as it shortens the drying time and cause minimum damage to the product. To reduce the energy utilization and operational cost new dimensions came up in drying techniques. Among the technologies osmotic dehydration, vacuum drying, freeze drying, superheated steam drying, heat pump drying and spray drying have great scope for the production of quality dried products and powders.

Keywords Superheated steam drying · Heat pump drying · Fruits and vegetable dehydration · Freeze drying · Spray drying · Pulsed electric field

Introduction

Fruits and vegetables are important sources of essential dietary nutrients such as vitamins, minerals and fibre. Since the moisture content of fresh fruits and vegetables is more than 80%, they are classified as highly perishable commodities (Orsat et al. 2006). Keeping the product fresh is the best way to maintain its nutritional value, but most storage techniques require low temperatures, which are difficult to maintain throughout the distribution chain. On the other hand, drying is a suitable alternative for post harvest management especially in countries like India where exist poorly established low temperature distribution and handling facilities. It is noted that over 20% of the world perishable crops are dried to increase shelf-life and promote food security (Grabowski et al. 2003). Fruits, vegetables and their products are dried to enhance storage stability, minimise packaging requirements and reduce transport weight. Nonetheless, in India hardly any portion of perishables are dried which leads to enormous loss in terms of money and labour besides steep rise in prices of commodities during the off season.

The preservation of fruits and vegetables through drying dates back many centuries and is based on sun and solar drying techniques. The poor quality and product contamination lead to the development of alternate drying technologies (Bezyma and Kutovoy 2005). The most applicable method of drying includes freeze, vacuum, osmotic, cabinet or tray, fluidized bed, spouted bed, Ohmic, micro wave and combination thereof (George et al. 2004). Except for freeze drying, applying heat during drying through conduction, convection and radiation are the basic techniques used to force water to vapourise, while forced air is applied to encourage the removal of vapour. A large number of food and biomaterials are dehydrated in a variety of units with diverse processing conditions. The choice of drying method depends on various factors such as the type of product, availability of dryer, cost of dehydration and final quality of desiccated product. Energy consumption and quality of dried products are other critical parameters in the selection of a drying process. To reduce the use of fossil fuel, electrical energy is an alternate source of energy for drying applications especially where electricity is generated by a renewable energy source such as hydro power or wind power (Raghavan and Orsat 1998, Raghavan et al. 2005). Keeping these in view, the present review is focussing on recent developments in drying and dehydration and future scope for better drying.

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Drying of fruits and vegetables

Drying of fruits and vegetables has been principally accomplished by convective drying (Nijhuis et al. 1998). There are a number of studies that have addressed the problems associated with conventional convective drying. Some important physical properties of the products have changed such as loss of colour (Chua et al. 2000), change of texture, chemical changes affecting flavour and nutrients and shrinkage (Mayor and Sereno 2004). Besides, convective drying gives little scope for prior rehydration to further processing after drying for a minimal quality (Khraisheh et al. 2004). The high temperature of the drying process is an important cause for loss of quality. Lowering the process temperature has great potential for improving the quality of dried products (Nindo et al. 2003, Beaudry et al. 2004). However in such conditions, the operating time and the associated cost become unacceptable. To reduce the operational cost different pre-treatments and new method of low temperature and low energy drying methods are evolved. A brief review of recent development (past 15 years) will be discussed in the following sections.

Osmotic dehydration (OD)

Osmosis is known as a partial dehydration process. Although it does not remove enough moisture to be considered as a dried product, the process has the advantage of requiring little energy. It works well as a pre-treatment prior to drying by other methods. The application of OD to fruits and to a lesser extent to vegetables, has received attention in recent years as a technique for production of intermediate moisture foods or as a pre-treatment prior to drying in order to reduce energy consumption or heat damage (Jayaraman and Gupta 1992).

Some aspects of osmotically dehydrated fruits have been reviewed by various workers with reference to osmotic agents and their concentration (Bolin et al. 1983), temperature (Le Maguer 1998), sample to solution ratio (Conwoy et al. 1983), agitation of fruit in syrup (Hawkes and Flink 1978), sample size and shapes (Islam and Flink 1982, Lerici et al. 1985), osmotic agents (Lenart and Flink 1984), material type (Talens et al. 2000), pre-treatment (Fito et al. 2001), size and shape (Lerici et al. 1985), temperature and concentration (Lazarides et al. 1995, Sagar and Kumar 2007), dehydration method and physico-chemical changes (Conwoy el aI 1983, Lenart and Flink 1984, Le Maguer 1998, Kumar et al. 2006).

Nsonzi and Ramaswamy (1998) modelled the mass transfer process with respect to moisture loss and solids gain. They stated that even though the moisture loss and solids gain occurred at the same time, the rate of moisture loss was much higher than the rate of solids gain. The advantage of OD is its lower energy use and lower product thermal damage since lower temperatures used allow the retention of nutrients (Shi et al. 1997). Lenart (1996) described the main advantages of using OD as the reduction of process temperature, sweeter taste of dehydrated product, reduction of 20–30% energy consumption and shorter drying time. Sagar and Kumar (2007) in OD of guava slices found that higher sugar concentration (60^0B) and temperature (60°C) increase the water loss from the produce and solid gain into the osmosed guava slices.

The driving force for the diffusion of water from the tissue into the solution is provided by higher osmotic pressure of hypertonic solution. The rate of mass transfer during OD is generally low. Techniques to improve mass transfer are partial vacuum (Rastogi and Raghava Rao 2004), ultra high hydrostatic pressure (Rastogi and Niranjan 2008), high intensity electrical field pulses (Rastogi et al. 1999), super critical CO_2 treatment (Tedjo et al. 2002) and prior to OD processing and using centrifugal force (Azura et al. 1996).

Vacuum

The reduction in pressure causes the expansion and escape of gas occluded into pores. When the pressure is restored, the pores can be occupied by the osmotic solution, increasing the available mass transfer surface area. The effect of vacuum application during OD is explained on the basis of osmotic transport parameter, the mass transfer co-efficient and the interfacial area (Rastogi and Raghava Rao 2004). Vacuum pressure (50-100 mbar) is applied to the system for shorter time to achieve the desired result.

High hydrostatic pressure

It is observed that application of high hydrostatic pressure damages the cell wall structure which leads to significant changes in the tissue architecture, leaving the cells more permeable, resulting in increased mass transfer rates during OD (Rastogi and Niranjan 2008).

Pulsed electric field (PEF)

The PEF treatment has been reported to increase the permeability of plant cells. PEF treatment-induced cell damage, resulted in tissue softening, which in turn resulted in a loss of turgor pressure, leading to a reduction in compressive strength. The increase in permeability of potato (Azura et al. 1996) and carrot (Rastogi et al. 1999) tissues by PEF treatment resulted in improved mass transfer during OD. The effective diffusion coefficients of water and solute increased exponentially with electric field strength. The increase in effective diffusion coefficient can also be attributed to an increase in cell wall permeability, which facilitated the transport of water and solute. Taiwo et al. (2002) studied the effect of PEF and other pre-treatments on OD of apple slices. It was concluded that PEF treatment increased water loss, which was attributed to increased cell membrane permeability. The effect of PEF treatment on solid gain was minimal. Lai and Sharma (2005) reported that PEF pretreatment (number of pulses 100, pulse width 840 μs, field strength 2.67 kV/cm, 1 Hz; specific energy input 3.58 kJ/kg) resulted in higher moisture loss and solid gain in mangoes during subsequent OD. Ade-Omowaye et al. (2003) compared the quality characteristics of PEF-pre treated and osmotically-treated red paprika with osmotic treatment at higher temperature. The retention of ascorbic acid and carotenoids was higher for PEF pre-treated and osmotically-treated paprika. Drying time of PEF-pre treated paprika was reduced by 25%. Taiwo et al. (2003) studied the influence of high intensity electric field pulses and osmotic dehydration on the rehydration characteristics of apple slices at different temperatures. Rehydration rate increased with temperature but higher rehydration capacity values were obtained at low temperatures (24°C and 45°C).

Super critical CO_2

This emerged as an attractive unit operation for the processing of food and biological material. The critical point of CO_2 gas is at 304.17 K and 7.38 MPa (Tedjo et al. 2002). The combination of pressure and temperature as process parameters makes it possible to vary the solvent of the medium within certain ranges as desired without having to change the composition of solvent. This treatment will not improve the water loss but favour solid gain.

Ultrasound

Acoustic streaming can affect the thickness of boundary layer which exists between stirred fluid and solid. Cavitation, a phenomenon produced by the sonication, consists of the formation of bubbles in the liquid which can collapse and generate localised pressure fluctuation. This ultimately increases the mass transfer of osmotic treatment. The rate of transfer depends on pressure and frequency of the wave produced by sonication (Raghavan et al. 2005).

Centrifugal force

Azura et al. (1996) applied a centrifugal force (64 g) during OD and obtained enhanced mass transfer of 15%. But prior work in variables like solvent, solute, plant tissue, permeability of tissue, mass transfer rate and solid gain are necessary to make the unit operation more successful.

Microwave heating

Microwave technology uses electromagnetic waves that pass through material and cause its molecules to oscillate generating heat. Microwave heating generates heat within the material and heats the entire volume at about the same rate. Microwave technology can be combined with conventional heating and drying units and is easily automated. The overall ratio of moisture loss to solid gain was higher in microwave assisted OD than in conventional OD (Xian-Ju et al. 2007).

Heat pump drying

The use of heat pumps for drying has been studied since the early 1950s; though the idea was mechanically feasible, it was not economically attractive due to the low fuel prices prevailing at that time. But the high fuel costs of the early 1970s revived the interest in use of heat pumps for drying due to the deemed energy savings. Conventional dryers would heat the air by using high quality energy (such as electricity or fuels) and vent a stream of moist, hot air at the exhaust, which represented a significant quantity of low-grade energy being lost from the process. In order to reduce this loss, heat pumps were introduced to the systems to recover the latent heat of evaporation of water lost in the exhaust from the dryer. By placing the evaporator of a heat pump in the exhaust stream , the air leaving the dryer is cooled (thereby recovering the sensible heat component) and then dehumidified (to recover the latent heat) by the refrigerant. The heat thus added to the refrigerant is then rejected at the condenser of the heat pump to the stream of air entering the dryer, thus raising its temperature. When the air leaving the dryer is recirculated, the added benefit of dehumidification of the drying air is also realised, increasing its potential to achieve better drying.

Hogan et al. (1983) studied heat pump assisted grain drying and concluded that the systems were useful due to their lower energy consumption in comparison with electrically heated units. Essentially, heat pumps were used to reduce the energy consumption of dryers and it is now universally agreed that heat pump dehumidifier (HPD) assisted drying systems do have energy savings (Queiroz et al. 2004, Seco et al. 2004). One of the main advantages of HPD drying is the retention of quality and its successful application to drying highly valued heat sensitive materials.

Kohayakawa et al. (2004) describe a study in which mango slices were dried in a HPD dryer and the energy consumption of this system was compared with a hypothetical electrically heated dryer. It is reported that the HPD dryer has an advantage of 22 to 40% reduction in the power consumption. Hawlader et al. (2006) dried apple, guava and potato pieces in a HPD dryer using nitrogen and carbon dioxide to replace air. The evacuation during drying seemed to benefit the final colour of the products, which showed lesser browning. Besides, the porosity and rehydration characteristics of the product were superior compared with material dried under vacuum. Gabas et al. (2004) studied drying of apple cylinders in a heat pump dryer and compared it with products obtained from an electrically heated dryer. The HP dryer used 40% less energy compared with the electrical heater and at a faster rate of drying. Alves-Filho et al. (2004) dried green peas in a fluidised bed heat pump dryer under atmospheric freeze-drying conditions and obtained products with high levels of rehydration ability, floatability and desirable colour characteristics. Uddin et al. (2004) compared heat pump drying with microwave and freeze drying of guava, mango and honeydew melon.

They suggested that microwave application be coupled with heat pump drying for better results such as faster drying rate, lower shrinkage, and better appearance of the product and low cost of the process.

Microwave drying

Microwave drying uses electrical energy in the frequency range of 300 MHz to 300 GHz, with 2,450 MHz being the most commonly used frequency. Microwaves are generated inside an oven by stepping up the alternating current from domestic power lines at a frequency of 60 Hz up to 2,450 MHz. A device called the magnetron accomplishes this (Orsat et al. 2005). The use of microwave energy for drying has been demonstrated to have a moderately low energy consumption (Tulasidas et al. 1995a). The volumetric heating and reduced processing time make microwaves an attractive source of thermal energy. Since microwaves alone cannot complete a drying process, it is recommended to combine techniques, such as forced air or vacuum, in order to further improve the efficiency of the microwave process (Chou and Chua 2001).

When the material couples with microwave energy, heat is generated within the product through molecular excitation. The critical next step is to immediately remove the water vapour. A simple technique for removing water is to pass air over the surface of the material hence combining processes to form what is called “microwave convective drying”. The air temperature passing through the product can be varied to shorten the drying time. In order to control the product’s temperature, either power density (Watts/g of material) or duty cycle (time of power on/off) must be controlled (Changrue et al. 2004). It is mainly in the falling rate period that the use of microwaves can prove most beneficial. As the material absorbs the microwave energy, a temperature gradient occurs where the centre temperature is greater, forcing the moisture out (Erle 2005). It is clear that the drying takes place in the falling rate period (Soysal et al. 2006). The dielectric loss factor of a material is a measure of the ability of material to dissipate electric energy. It is important to realise that dielectric properties are specific only for a given frequency and materials’ properties. The dielectric properties change as a function of temperature and moisture, hence the uniformity of moisture and drying temperature govern the uniformity of the drying process (Meda and Raghavan 2004).

Use of microwave energy in drying offers reduced drying times and complements conventional drying in later stages by specifically targeting the internal residual moisture (Osepchuk 2002). The drying of banana slices with microwave demonstrated that good quality dried products can be achieved by varying power density and duty cycle time. In dried carrots, quality improvement was found in colour, shrinkage and rehydration property (Wang and Xi 2005). The quality of raisins dried by microwave was superior to hot air dried samples in colour, damage, darkness, crystallised sugar, stickiness and uniformity (Tulasidas et al. 1995b).

Superheated steam drying

Drying with superheated steam (SS) in the absence of air in a medium composed entirely of steam. The ability of SS to dry food material is due to the addition of sensible heat to raise its temperature above the corresponding saturation temperature at a given pressure. It is not necessary to exhaust the evaporated water from the produce until the pressure develops beyond certain limit. After that excess steam will be released. The great advantage is that recycling of drying method is possible, provided additional sensible heat is added. Besides, any conventional convection and conduction dryer could be easily converted to use superheated steam (Tatemoto et al. 2007). Fixed bed, fluidized bed, flash, impingement, pneumatic and spray dryers are using superheated steam technology for quality drying of produces.

SS fluidized drying

Trials indicate that SS drying could be effectively used for many products like corn starch, potato starch and for making other by products. However, particles that are too large or fine produces are impossible to dry in a fluidised bed. The model was developed based on diffusivity theory and uses a number of assumptions. Among them are: i) condensation of water vapour on samples occurs below the boiling point of water, ii) all of the heat transferred into the sample surface is used for evaporation when the sample temperature is equal to the boiling point, iii) boiling point of water changes the pressure in the local point of sample, iv) overall heat transfer coefficient on the sample surface includes thermal radiation from the drying medium and v) drying process is complete when the temperature of sample is higher than the boiling point of water (Tatemoto et al. 2007).

Impingement drying with SS

Though it is mainly used in paper industries, in the food industry, air impingement is used for baking and cooking of products such as potato chips, pizza, cookies and flat breads (Rahman and Labuza 1999). Low fat potato chips can be prepared by this method. SS processed potato chips retained more vitamin C and were better in texture than air dried samples. It is observed that mass transfer was following Ficks law of diffusion and heat transfer within potato was considered to follow Fouriers law of conduction (Leeratamark et al. 2006). However, attention should be given to the effect of SS impingement drying on product quality including shrinkage, crispness and microstructure.

SS flash drying

Food products sensitive to high temperature have a high potential to be processed with SS flash drying when processing is under vacuum. Pneumatic or flash drying is a process in which the transported gas changes into SS. It is mainly used to dry organic compounds like lignite, bark, peat and pulp (Okos et al. 1992).

Freeze drying

Freeze drying of biological materials is one of the best methods of water removal which results in final product of the highest quality. Freeze drying is sublimation of ice fraction where water passes from solid to gaseous state. Due to very low temperature all the deterioration activity and microbiological activity are stopped and provide better quality to the final product. Recently the market for organic products is increasing. Therefore, the use of freeze drying of fruits and vegetables is not only increasing in volume but also diversifying (Brown 1999). Freeze drying seems to be better preservation method over other dehydration methods such as air or drum drying (Hsuch et al. 2003). Nonetheless, freeze drying of small fruits (strawberry) received particular attention by several researchers (Paakkonen and Mattila 1991, Hammami and Rene 1997, Shishegarha et al. 2002). Strawberry dried at 20°C retained better quality than at 60°C. The product mostly collapses i.e. loss in structure, reduction in pore size and shrinkage at higher temperature (Hammami and Rene 1997). Paakkonen and Mattila (1991) have found that low processing temperature improved the sensory quality of dried fruits.

Spray drying (SD)

The SD is a well known industrial technology used extensively on a large scale for drying and powdering heat sensitive materials from liquid foods. The overall objective in SD is to get the most rapid liquid removal with minimal negative impact on the product, without damaging the surrounding environment at the lowest capital and operating costs (Hall 1996). By using heat, SD efficiently transforms a dilute fluid suspension into a dry powder and renders good quality to final powder (Masters 1991). SD process comprises of 4 basic steps (Masters 1991, Filkova and Majumdar 1995): i) atomisation, ii) contact between drop lets and hot gas, iii) water evaporation and iv) gas-powder separation. Uniformity of drop size and homogeneity of spray jet are important considerations in designing nozzle. Pneumatic two-fluid nozzle, pressure nozzle and cone nozzle are most commonly used. Drying through SD may either in single stage, 2 or 3 stages. Pneumatic nozzle type driers mostly worked with single stage drying. Two-stage system comprises of the spray dryer followed by a vibro-fluidized bed system. Three stage processes improve the properties of dried powder by instant reconstitution because the fluid bed works as a dryer-agglomerator, controlling particle agglomeration (Master 2004).

Vacuum drying

Vacuum drying is an important process for heat sensitive materials. The process of vacuum drying can be considered according to physical condition used to add heat and remove water vapour. Low temperature can be used under vacuum for certain methods that might discolour or decompose at high temperature. A comparison of drying technologies in review by Khin et al. (2005) showed that freeze drying, vacuum drying and osmotic dehydration are considered too costly for large scale production of commodity.

Hybrid drying/Combination drying

Hybrid drying techniques are becoming common since the combined technology receives the benefits of individual process. Combination drying systems were tested by Feng et al. (1999). The number of combinations possible is vast and as technology continues to improve more will be developed. Adding a micro wave system to a spouted bed system combines the benefits offered by each technology. The microwave action decreases drying time while the fluidization produce by the spouting system improves drying uniformity, thus reducing the burning. Thermal-vacuum dryer intended for drying agricultural products can be manufactured with cheap and widely used materials such as wood or plastic.

Chou and Chua (2001) reviewed new hybrid drying technologies for heat sensitive food. Donsi et al. (1998) showed the combination of hot air drying and freeze drying increased the quality of dehydrated fruits and vegetables. Kumar et al. (2001) showed the combination process produced dehydrated carrot and pumpkin having similar quality as freeze dried products. The drying time and total energy consumption was favourably 50% lower than freeze drying alone.

High initial cost, loss of aroma, degradation of texture are some of the disadvantages of microwave drying. Combination drying with an initial conventional drying process followed by a finish microwave or microwave vacuum process has proven to reduce drying time while improving product quality and minimising energy requirements (Erle 2005, Soysal et al. 2006).

Microwave convection and microwave vacuum drying

Since the boiling point of water gets reduced at lower pressures, vacuum can be applied to microwave drying to improve product quality. There have been numerous studies on the application of vacuum to microwave drying. Drouzas and Schubert (1996) investigated vacuum-microwave drying of banana slices. The product quality in terms of taste, aroma, and rehydration was found to be excellent. Tein et al. (1998) compared dried carrot slices using vacuum/microwave drying with air and freeze drying. Microwave vacuum dried carrot slices had higher rehydration potential, higher β-carotene and vitamin C content, lower density and softer texture than those prepared by air drying. Similar results were reported by Regier et al. (2005), where microwave vacuum dried carrots had the highest carotenoid retention compared with freeze drying and convection drying.

Combined process consisting of an initial air drying step followed by a microwave-vacuum process for producing dried fruits and vegetables of high value. Pre drying to a moisture content of 25–40% wet weight basis (wwb) is carried out in a microwave-vacuum dryer. Dehydration to a final moisture content of 4–6% (wwb) can be executed in continuous air-band dryer or in a moving tray dryer to boost the capacity of microwave-vacuum dryer (Beaudry et al. 2003). Pulsed-microwave-vacuum drying is suitable for temperature sensitive produces (Gunasekaran 1998).

A comparative study was conducted by Cui et al. (2003) for garlic drying. Best quality dried garlic was obtained with freeze drying and microwave vacuum drying as a close second while there was a great loss in garlic pungency with the hot air dried samples. Sharma and Prasad (2006) came to similar conclusion while comparing hot air drying with microwave convective drying of garlic. They obtained a drying time reduction of 80% with superior quality dried garlic by combining microwaves at 0.4 W/g to hot air at 60–70°C.

Microwave osmotic dehydration

There are some food products for which the skin impedes water transport to the surface. In these cases, a pre-treatment is required before osmosis as was described for the pre-treatment of cranberries (Sunjka and Raghavan 2004). Beaudry et al. (2004) studied the effect of microwave convective (0.7W/g and 62°C), hot air (62°C), freeze and vacuum drying (94.6 kPa) methods on the quality of osmotically dehydrated cranberries. With all drying methods, they reported that the constant drying rate period is no longer present following osmotic dehydration. This effect was also reported by Piotrowski et al. (2004) with strawberries. The fastest drying method was microwave convective, and the longest was for hot air drying at 62°C. Venkatachalapathy and Raghavan (1999) reported that combined osmotic microwave dried strawberry was close to that of freeze-dried product in terms of rehydration characteristics and overall sensory evaluation.

Quality attributes and classification

There are several changes taking place in quality parameters during drying and storage. The extent of changes depends on the care taken in preparing the material before dehydration and on the process used. Major quality parameters associated with dried food products include colour, visual appeal, shape of product, flavour, microbial load, retention of nutrients, porosity-bulk density, texture, rehydration properties, water activity, freedom from pests, insects and other contaminants, preservatives, and freedom from taints and off-odours (Ratti 2005). The state of the product, such as glassy, crystalline or rubbery, is also important. These quality parameters can be classified into 4 major groups: i) physical, ii) chemical, iii) microbial and iv) nutritional. Greater stability and quality can be achieved by maintaining the fresh or optimum conditions of the raw materials (Perera 2005).

Physical quality

Physical changes, such as structure, case hardening, collapse, pore formation, cracking, rehydration, caking and stickiness can influence the quality of final dried products. Hot air drying usually destroys the cell structure and thereby takes more time for dehydration while due to solid state of water during freeze drying protects primary structure and shape of products keeping the cells almost intact, with a high porosity end products (Saguy et al. 2004). Pretreatments given to foods before drying or optimal drying conditions are used to create a more porous structure so as to facilitate better mass transfer rates. Maintaining moisture gradient levels in the solid, which is a function of drying rate, can reduce the extent of crust formation; the faster the drying rate, the thinner the crust (Achanta and Okos 1996). Depending on the end use, hard crust and pore formation may be desirable or undesirable. Rahman (2001) has outlined the current knowledge on the mechanism of pore formation in foods during drying and related processes. The glass transition theory is one of the concepts proposed to explain the process of shrinkage and collapse during drying and other related processes. According to this concept, there is negligible collapse (more pores) in a material when it is processed below the glass transition. The higher the process temperature above the glass transition temperature (Tg), the higher the structural collapse. The methods of freeze and hot air drying can be compared based on this theory. In freeze-drying, since the temperature is below Tg' (maximally freeze concentrated Tg), the material is in the glassy state. Hence shrinkage is negligible. As a result the final product is very porous. In hot air drying, on the other hand, since the temperature of drying is above Tg' or Tg, the material is in the rubbery state and substantial shrinkage occurs. Hence the food produced from hot air drying is dense and shrivelled (Peleg 1996, Sablani and Rahman 2002).

Recent experimental results show that the concept of glass transition may not be valid for freeze-drying of all types of biological materials, indicating the need for incorporation of other concepts such as surface tension, structure, surrounding pressure and mechanisms of moisture transport (Sablani and Rahman 2002, Meda and Ratti 2005). Rahman (2001) hypothesised that since capillary force is the main factor responsible for collapse, then counter balancing this force will cause formation of pores and lower shrinkage. This counterbalancing is due to generation of internal pressure, variation in moisture transport mechanism and surrounding pressure. Another factor could be the strength of the solid matrix (ie, ice formation, case hardening and matrix reinforcement). Quality parameters such as volume, shrinkage, apparent density, colour and rehydration behaviour of carrots dried in SS under vacuum were superior to air vacuum dried carrots. Starch gelatinization in potato slices occurred more rapidly in SS drying than in hot air drying, leading to glossy appearance on the surface. Basil leaves dried in SS under vacuum, rendered a product with a higher retention of the original volatile compounds than conventional air drying (Lovedeep et al. 2002).

Rehydration is the process of moistening a dry material. In most cases, dried foods are soaked in water before cooking or consumption, thus rehydration is one of the important quality criteria. In practice, most of the changes during drying are irreversible and rehydration cannot be considered simply as a process reversible to dehydration (Lewicki 1998). In general, absorption of water is rapid at the beginning. A rapid moisture uptake is due to surface and capillary suction. Rahman and Perera (1999) and Lewicki (1998) reviewed the factors affecting the rehydration process. These factors are porosity, capillaries and cavities near the surface, temperature, trapped air bubbles, amorphous-crystalline state, soluble solids, dryness, anions and pH of the soaking water. Porosity, capillaries and cavities near the surface enhance the rehydration process, whereas the presence of trapped air bubbles give a major obstacle to the invasion of fluid. When the cavities are filled with air, water penetrates to the material through its solid phase. In general, temperature strongly increases water rehydration in the early stages. There is a resistance of crystalline structures to salvation that causes development of swelling stresses in the material, whereas amorphous regions hydrate fast. The presence of anions in water affects the volume increase during water absorption (Sablani 2006b).

Texture of dried products are influenced by their moisture content, composition, pH, and product maturity. The chemical changes associated with textural changes in fruits and vegetables include crystallisation of cellulose, degradation of pectin, and starch gelatinisation. In meat products the changes such as aggregation and denaturation of proteins and a loss of water-holding capacity leads to toughening of muscle tissue. The method of drying and process conditions also influence the texture of dried products. Krokida et al. (2001) studied the quality of apple, banana, potato and carrot with different drying methods such as convective, vacuum, microwave, freeze and osmotic drying. It was found that air, vacuum and microwave dried materials caused extensive browning in fruits and vegetables whereas freeze drying seemed to preserve colour changes, resulting a produce with improved colour characteristics.

Chemical quality

Browning, lipid oxidation, colour loss and change of flavour in foods can occur during drying and storage. Browning reactions can be categorised as enzymatic and non-enzymatic (Salunkhe et al. 1991). Enzymatic browning of foods is undesirable because it develops undesirable colour and produces off flavour. The application of heat, sulphur dioxide or sulphites and acids can help control this problem. The major disadvantage of using these treatments for food products is their adverse destructive effect on vitamin B or thiamine. Acids such as citric, malic, phosphoric and ascorbic are also employed to lower pH thus reducing the rate of enzymatic browning. Dipping in osmotic solution can inhibit enzymatic browning in fruits. This treatment can also reduce the moisture content with osmotic pre-concentration. There are three major types of non enzymatic reaction: (i) Maillard reaction, (ii) caramelisation and (iii) ascorbic acid oxidation (Salunkhe et al. 1991). Factors that can influence non-enzymatic browning are water activity, temperature, pH and the chemical composition of foods. Browning tends to occur primarily at the mid-point of drying period. This may be due to migration of soluble constituents towards the centre. Browning is also more severe near the end of the drying period when the moisture level of the sample is low and less evaporative cooling is taking place.

Rapid drying through 15–20% moisture range can minimise the time for Maillard browning. In carbohydrate foods, browning can be controlled by removing or avoiding amines and conversely, in protein foods, by eliminating the reducing sugars. Sulphur treatment can prevent the initial condensation reaction by forming nonreactive hydroxy-sulphonate sugar derivatives. In caramelisation, heating of sugars produces hydroxy methyl furfural, which polymerises easily. This reaction may be slowed by sulphite, which reacts with sugars to decrease the concentration of the aldehydic form. Discolouration of ascorbic acid containing vegetables can occur due to formation of dehydroascorbic acid and diketogluconic acids from ascorbic acid during the final stages of drying. Sulphur treatment can prevent this browning due to reactivity of bisulphite towards carbonyl groups present in the breakdown products (Kadam et al. 2008).

Fatty foods are prone to develop rancidity at very low moisture content (less than monolayer moisture). Lipid oxidation is responsible for rancidity, development of off-flavours, and the loss of fat-soluble vitamins and pigments in many foods, especially in dehydrated foods. Factors that influence the oxidation rate include moisture content, type of substrate (fatty acid), extent of reaction, oxygen content, temperature, presence of metals or natural antioxidants, enzyme activity, UV light, protein content and free amino acid content. Moisture content plays a big part in the rate of oxidation. Air-dried foods are less susceptible to lipid oxidation than freeze-dried products due to lower porosity (Sablani 2006a).

Microbial quality

Dried food products are considered safe with respect to microbial hazard. There is a critical water activity (a_w) below which no micro organisms can grow. Pathogenic bacteria cannot grow below a_w of 0.85–0.86, whereas yeast and moulds are more tolerant to a reduced water activity of

0.80. Usually no growth occurs below a_w of about 0.62 (Sablani 2006a). Reducing the water activity inhibits microbial growth but does not result in a sterile product. The heat of the drying process does reduce total microbial count, but the survival of food spoilage organisms may give rise to problems in the rehydrated product. The type of microflora present in dried products depends on the characteristics of the products, such as pH, composition, pre-treatments, types of endogenous and contaminated microflora and method of drying. Brining (addition of salts) in combination with drying decreases the microbial load. The dried products should be stored under appropriate conditions to protect them from infection by dust, insects and rodents (Rahman et al. 2000).

Nutritional quality

Fruits, vegetables and their products in the dried form are good sources of energy, minerals and vitamins. However, during the process of dehydration, there are changes in nutritional quality (Sablani 2006a). A more number of vitamins such as A, C and thiamine are heat sensitive and sensitive to oxidative degradation. Sulphuring can destroy thiamine and riboflavin while pre-treatments such as blanching and dipping in sulphite solutions reduce the loss of vitamins during drying. As much as 80% decrease in the carotene content of some vegetables may occur if they are dried without enzyme inactivation. However, if the product is adequately blanched then carotene loss can be reduced to 5% Steam blanching retains higher amounts of vitamin C in spinach compared with hot-water blanching (Ramesh et al. 2001). Blanching in sulphite solution can retain more ascorbic acid in okra (Inyang and Ike 1998). Na-metabisulphite treatment was able to reduce oxidation of carotenoid in carrots and L-cysteine-HCl help in retaining highest amount of ascorbic acid (Mohamad and Hussein 1994). SS fluidized bed processing was used for both drying and inactivation of antinutritional factors trypsin inhibitor and urease in soybean (Piotrowski et al. 2004).

The retention of vitamin C in freeze-dried products is significantly higher than that of oven and sun-dried products. Microwave and vacuum drying methods can also reduce the loss of ascorbic acid due to low levels of oxygen. Microwave, Refractance Window, low pressure superheated steam and vacuum drying can also reduce the loss of vitamins due to a low level of oxygen. Shade drying, in the absence of light, can also be effective for the retention of nutrients (Sablani 2006a).

Sensory properties of dried foods are also important in determining quality. These include colour, aroma, flavour, texture and taste. Aroma and flavour can change due to loss of volatile organic compounds, the most common quality deterioration for dried products. Low temperature drying is used for foods that have high economic value such as flavouring agents, herbs and spices (Salunkhe 1991, Singh et al. 2006). Low temperature drying is important for heat sensitive products. Singh et al. (2006) found that low temperature dryer caused minimal damages to leafy vegetables during drying and thereby retained more nutrients than other dryers. Processing in the absence of oxygen can preserve many components which are sensitive to oxidation (Feng et al. 1999). Microwave drying can reduce the drying time and improve the quality (Beaudry et al. 2003). A study by Zhong and Lima (2003) showed that Ohmic heating accelerated the vacuum drying rates of sweet potato.

Influence on storage on quality

A significant loss of nutrients occurs in dried fruits and vegetables during storage. This loss depends on storage temperature, pH, exposure to oxygen, porosity, light and presence of organic acids. The extent of losses depends on the type of vitamins and storage conditions such as the exposure to oxygen and light (Sablani 2006a). During storage of spaghetti, for example, no loss of thiamine and niacin was observed but riboflavin was susceptible to temperature, storage period and light (Watanabe and Ciacco 1990).

In some situations the method of dehydration can also influence the loss of nutrients. For instance, Kaminski et al. (1986) observed a rapid degradation of carotenoids in freeze-dried carrots. They observed that air-drying was more efficient for carotene preservation when stored at ambient temperature. Freeze-dried products are generally more porous. This facilitates oxygen transfer and promotes rapid oxidation of carotene. Cinar (2004) reported that the highest pigment loss was in carrot stored at 40°C (98.1%) while the lowest loss was in sweet potato kept at 4°C (11.3%) during 45 days of storage.

Energy efficiency in drying

Drying is commonly employed to prevent the post harvest deterioration of many fruits and vegetables (Beker 2005). In most cases, some form of through-flow or cross-flow convective dryer is used for this purpose. A common feature of these dryers is their high use of energy. There is considerable concern world wide over global warming which is attributed to green house gases produced by the combustion of fossil fuels. As a result there is increasing pressure to reduce energy consumption, particularly in those countries that are signatories to Kyoto protocol. Raghavan et al. (2005) indicated that about 34% of the world produce requires artificial drying at least for part of the crop.

Ways to reduce energy consumption

Routine care in operation should always form an integral part of crop-dryer operation. Combination drying, another best way to reduce the energy consumption, increases the through-put and improves quality (Raghavan et al. 2005). Optimization of energy through mathematical modelling is another important way to reduce energy consumption (Achariyaviriya et al. 2002). Intermittent drying (Chua et al. 2003) and electro drying technologies (Raghavan et al. 2005) are also used to reduce energy consumption. The application of microwave was found to have a major impact on both the drying time and the energy consumption. The specific energy consumption for the drying of grapes reduced from 81.15 MJ/kg in case of convective drying to 7.11–24.32 MJ/kg by combined microwave – convective drying (Tulasidas et al. 1995a). Wang et al. (2002) described mathematical model of the drying of 4 mm carrot particles in a batch fluidized bed dryer with microwave heating. Infrared-convective dryers reduce the energy consumption of osmotically pre-treated samples of potato and pineapple (Tan et al. 2001). Heat pump dryers and high electric field dryers have the great potential for industrial application, particularly for high value crops because of superior quality of its products, simplicity of design and low energy use (Regaldo et al. 2004).

Conclusion and future trends

Many new dimensions came up in drying technology to reduce the energy utilization and operational cost. Among the technologies, osmotic dehydration, vacuum drying, freeze drying, SS drying, HPD drying microwave drying and spray drying are offering great scope for the production of best quality dried products and powders. Due to their selective and volumetric heating effects, microwaves bring new characteristics such as increased rate of drying, enhanced final product quality and improved energy consumption. The quality of microwave dried commodities is often between air-dried and freeze-dried products. The rapidity of the process yields better colour and aroma retention. Quality is further improved when vacuum is used since the thermal and oxidative stress is reduced. Due to high cost, using single unit operation to dry the produce is not cost effective. Therefore, cost effective alternate systems like combination/hybrid drying should be promoted to reap the advantage of sophisticated drying systems with minimum cost and simple technologies. Combination drying with an initial conventional drying process followed by a microwave finish or microwave vacuum process has proven to reduce drying time while improving product quality and minimising energy requirements. However, several factors should be taken into consideration when developing drying system for the fruits and vegetables. An optimal drying system for the preservation of fruits and vegetables should be cost effective, shorter drying time and with minimum damage to the product. Researchers from different centres are focussing on mathematical modelling and computer simulation as important technology that can provide information on the process parameters that would otherwise be unavailable.

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Trends in quality assessment and drying methods used for fruits and vegetables

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ABSTRACT

Studies on drying of fruits and vegetables by means of different methods are numerous. This review aims at presenting the most usual quality analyses carried out in dried fruits and vegetables over the last five years along with the most used drying methods. It was prepared based on a search on Scopus database with the term 'drying fruit vegetables'. Results show that color and microstructure can be preserved, apparent density can be reduced, and high rehydration capacity can be achieved by using mild drying methods like freeze-drying and innovative drying methods like conductive multi-flash drying. Shrinkage and water activity can be minimized by using pretreatments like osmotic dehydration, ultrasound, and pulsed electric fields. Such pretreatments also produce crispy texture, which can also be obtained with the use of hot air drying followed by microwave-vacuum drying. Antioxidant activity, phenolic compounds and vitamin C content can be maximized by using innovative pretreatments like immersion in solutions rich in bioactive compounds and short drying time. Microbial counts can be decreased by using pretreatments like immersion in ethanol, and modified atmosphere packaging. High sensory acceptability can also be achieved by using innovative drying methods, like instant controlled pressure drop drying.

1. Introduction

Drying generally describes the process of producing a solid product by removing water by heating (Mujumdar, 2015). Many methods of food drying have been described. Fruits and vegetables are suitable materials for drying because of their short shelf life, which can extend their shelf life and create unique sensory characteristics (see Fig. 1).

Among the approaches used in drying studies, the product quality assessment is quite common. This is justified by the fact that it is necessary to know, besides aspects like process efficiency and modelling, if the yielded dried product presents an acceptable quality. In this sense, physicochemical, physical, chemical, sensory, and microbiological measurements can be performed.

Even though the studies on drying of fruits and vegetables are numerous, a search on Scopus database with the terms "dried fruit vegetables quality" returned 55 "review" papers, from which none of them dealt with the theme of this paper. For producing this review, our exclusion criteria were the following: we used the terms "drying fruit vegetables" and limited the search to "articles", and then we manually selected those articles in which some quality assessment in dried fruit and vegetables was effectively performed; articles form the last 5 years were selected. Finally, the studies selected were the ones in which pieces were produced as a final product, i.e., powders were excluded. Therefore, the aim of this review is to present the most common quality analyses performed in dried fruits and vegetables over the last five years. Furthermore, an overview of the drying methods used in those reports and their advantages and disadvantages for the quality of fruits and vegetables are presented.

2. Physical analyses

2.1. Instrumental color measurement

To see color, it is required a light source, an object, and an observer (Hunterlab, 2015). Color is the first attribute perceived by consumers in food. Color of dried fruit and vegetables can be influenced by reactions

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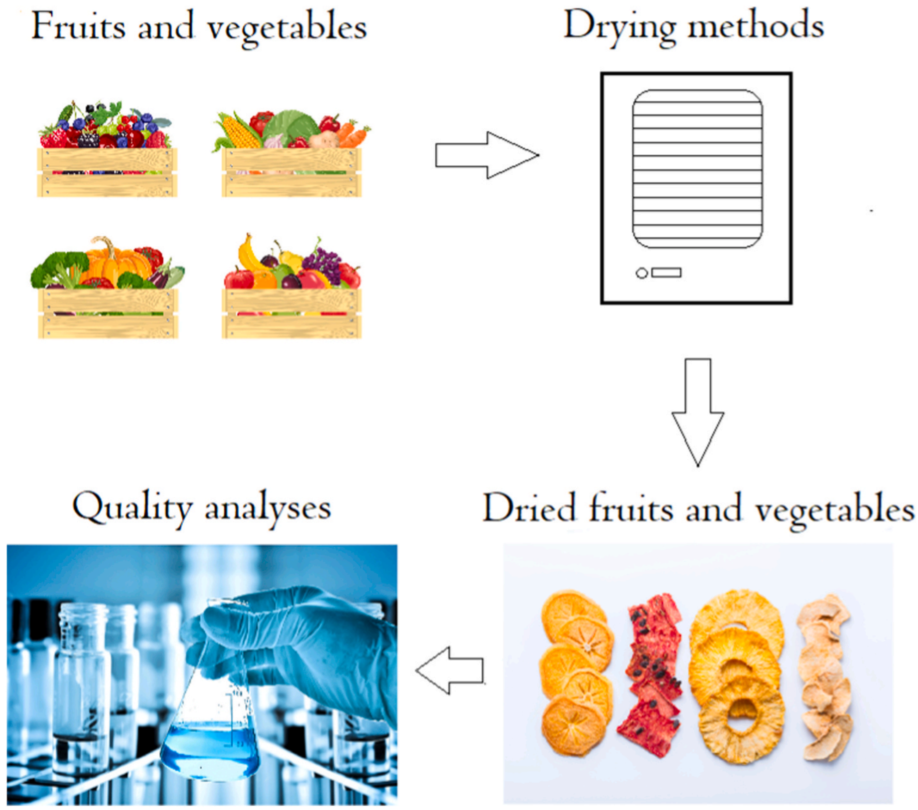

Fig. 1. Topics dealt with in this review.

like enzymatic browning and non-enzymatic browning (Richter Reis et al., 2017). Studies dealing with color measurement of dried fruit and vegetables show that the CIE L*a*b* color scale is commonly used for expressing their color (Kumar et al., 2021; Li et al., 2019; Al Maiman et al., 2021). Such scale contains the L (lightness) axis, in which 0 is black, 100 is white, and 50 is middle gray; the (red-green) axis, in which positive values are red, negative values are green, and 0 is neutral; and the b (blue-yellow) axis, in which positive values are yellow, negative values are blue, and 0 is neutral. It is usual to use the D_{65} illuminant, which represents noon daylight and the 10° observer, that best correlates with average visual assessments made with large fields of view, typical of most commercial applications. A parameter that is also very used is the total color difference, calculated as follows (Hunterlab, 2015):

$$\Delta E = \sqrt{\Delta L^*)\Delta a^*)(\Delta a^*)(\Delta a^*)^2 + \Delta b^*)^2} \qquad \text{(eq. 1)}$$

where L* is lightness, a* is redness, and b* is yellowness.

During microwave drying of okra, a decrease in L* was observed, which was interpreted as browning. A decrease in a* and b* values was also detected, which was considered to be a decrease in greenness due to decomposition of chlorophyll and carotenoids, along with the formation of Maillard reaction products (Aamir & Boonsupthip, 2017). Jujube slices processed by Pulsed Air-Jet Impingement Drying presented a decrease in L*, a* and b* with drying time, which was attributed to browning (Cao et al., 2018). Mango slices dried by conductive multi-flash drying (KMFD) with 14 heating vacuum pulse cycles and 2.55 kPa s^{-1} pressure drop rate and by conductive multi-flash drying with 4 cycles followed by vacuum drying (KMFD-VD) presented color closer to fresh mangoes, i.e., low values of total color difference (ΔE) (Link et al., 2018). A decrease in L*, a* and b* was observed for carrots pretreated by osmotic dehydration and dried by Instant Controlled Pressure Drop (DIC). These results were attributed to the degradation of carotenoids. However, the solid gain during osmotic dehydration helped to preserve the original color of carrots (Peng, Bi, et al., 2018).

Hot air drying, vacuum drying, and hot air drying followed by microwave vacuum drying produced dried kiwifruits with similar color (increased a* and b* and similar L* when compared to fresh sample), while freeze-drying yielded a product lighter color (Akar & Barutçu Mazı, 2019). Orange slices presented lower total color difference (ΔE) from fresh samples when dried by vacuum microwave drying, as compared to vacuum infrared drying and hot air drying (Bozkir, 2020). Apple slices dried by refractance window and hot air drying presented increase in L*, decrease in a* and increase in b*. In addition, the use of higher drying temperatures and osmotic dehydration coupled with moderate electric field increased the value of ΔE (from fresh samples) (Hernández et al., 2020). The use of sucrose and apple juice as osmotic solutions for Japanese quince chips led to lower values of ΔE (from fresh samples) and higher values of saturation (C*) when compared to chokeberry juice. With regard to drying methods, hot air drying caused browning, while freeze-drying led to the lower ΔE (Kowalska, Marzec, Domian, Galus, et al., 2020). The hot air drying of tomatoes was optimized toward color quality by Obajemihi et al. (2020), who found that the desirable (maximal) hue angle is 39.55° and the desirable (minimal) ΔE is 33.64. These values can be achieved by using 50 °C air temperature, 5.11 tomato slice thickness, *Tiwantiwa* tomato cultivar and pre-drying treatment of honey and sugar solution. An increase in ultrasound power during cold air drying (5 °C) followed by far-infrared radiation drying (120 °C) of potatoes led to lower ΔE (from fresh samples) (Zhang et al., 2020).

Yam snacks produced by infrared-assisted spouted bed drying presented higher L* and a* and lower b* when compared to fresh samples. This result was attributed to the presence of a starchy surface in the dried samples (Li et al., 2021). A decrease in L*, a* and b* was observed in solar dried tomatoes when compared to fresh samples. The result was attributed to decomposition of pigments and non-enzymatic browning (Al Maiman et al., 2021). Hot air drying of persimmons led to increase in a*, which was attributed to Maillard reaction. Blanching of persimmons attenuated this effect (Oshima et al., 2021).

Summarizing, the most common changes in fruit and vegetables color during drying are ascribed to browning reactions and natural pigments degradation, which can be attributed to the heat used for drying. When using blanching, osmotic dehydration and innovative and mild drying methods, there is an improvement in the color of dried fruit and vegetable pieces.

2.2. Apparent or bulk density

By definition, density is the mass of a solid per unit volume. However, in porous media like dried fruits and vegetables, more than one type of density can be defined. In the last years, apparent or bulk density was the property of choice for expressing density of fruit and vegetable pieces. It can be defined as:

$$\rho_b = \frac{m}{V_b} \quad \text{(eq. 2)}$$

where ρ_B is the apparent density (g.cm^{-3}), m is the sample mass (g) and V_b is the bulk volume (cm^{-3}). Dried products of high apparent density are considered of low quality, while dried products of high porosity and low apparent density are considered of high quality (Rodríguez-Ramírez et al., 2012). When conductive multiflash drying (KMFD) was combined with vacuum drying, the lowest values of apparent density were observed in mango slices when compared to KMFD alone (Link et al., 2018). Pineapples pretreated by osmotic dehydration, ultrasound and pectin-acid coating followed by hot air drying presented higher apparent density than control samples (without pretreatment) (Sakooei-Vayghan et al., 2020). Jujube slices presented lower apparent density when dried by freeze-drying, followed by pressure-differential puffing drying and hot air drying (Wang et al., 2021). Bananas dried in a hybrid-solar-vacuum dryer presented the lowest values of apparent density when experiments were carried out without the use of a vacuum reservoir (Roratto et al., 2021).

To sum up, innovative drying methods and freeze-drying yield dried fruit and vegetables with lower apparent density, i.e., higher quality than hot air drying. Low apparent density is usually related to higher porosity and crispness, which are desirable aspects of dried foods.

2.3. Microstructure

The knowledge of the microstructure of dried fruits and vegetables can provide important data for understanding their quality on macro scale. Studies on this theme have been using techniques like scanning electron microscopy (Farhaninejad et al., 2017; Hernández et al., 2020; Li et al., 2021; Roratto et al., 2021; Vallespir et al., 2018; Wang et al., 2021; Yang et al., 2019), transmission electron microscope (Zhang et al., 2018), light microscope (Zhou et al., 2021), and X-ray microtomography (μCT) scanning (Li et al., 2020; Peng et al., 2018a, 2018b) for obtaining the microstructural images.

The microstructure analysis of banana slices subjected to osmotic dehydration associated with ultrasound and mechanical agitation showed that control samples presented less pores and lower tissue damage, while mechanically agitated samples presented increased pores and tissue damage, and ultrasound treated samples presented the highest porosity and tissue damage (Farhaninejad et al., 2017). Longer osmotic dehydration times led to higher number of large pores in carrots dried by DIC, which was attributed to degradation of carrot polymers by the heat of osmotic pretreatment (Peng, Bi, et al., 2018).

Carrots pretreated by freezing in liquid N_2 and dried by DIC showed presented uniformly distributed and smaller pores than carrots frozen at −18 °C, −40 °C and −80 °C, suggesting that the former presented a desirable crispy texture (Peng, Yi, et al., 2018). Similar results were found for beetroots, apples, and eggplants (Vallespir et al., 2018). The microstructure of vacuum impregnated chestnut slices was better preserved by freeze-drying and vacuum-drying when compared to hot air drying. Hot air-dried samples presented browning and folds, which was attributed to the high temperatures achieved during the process (Yang et al., 2019).

Apple slices pretreated by osmotic dehydration coupled with moderate electric field and dried by hot air drying (control) and refractance window presented a less collapsed structure due to pretreatment, in the case of hot air drying, and an open structure with the presence of more pores for both pretreated and unpretreated samples, in the case of refractance window (Hernández et al., 2020). The use of hot air drying followed by moisture equilibrium process (MEP) and DIC provided apple cubes with a homogeneous pore size that resulted in a better expansion behavior (Li et al., 2020). MEP consisted in storing the apple cubes at 0.4 g g^{-1} relative humidity for 48h before DIC.

Yam cubes impregnated with probiotic bacteria and dried by infrared-assisted spouted bed drying presented microstructural images that showed the starch granules and the bacteria adhered to them, suggesting that the drying process used is a viable way for producing probiotic yam (Li et al., 2021). The microstructure of bananas dried in a hybrid-solar-vacuum dryer was better preserved when only one vacuum pulse was used, resulting in a higher number of pores and a crispy texture (Roratto et al., 2021). Pressure-differential puffing drying (PPD) was the best method for preserving the original microstructure of jujubes when compared to freeze-drying and hot air drying. When dried by PPD, jujubes presented a honeycomb-like microstructure (Wang et al., 2021).

In sum, innovative methods like ultrasound assisted osmotic dehydration, DIC, refractance window, infrared-assisted spouted bed drying, and pressure-differential puffing drying were more favorable for yielding a good microstructure from a sensory perspective and for producing probiotic vegetables.

2.4. Rehydration

Rehydration consists in moistening dry material. Usually, it is done by abundant amount of water. In general, the higher the temperature, the higher the increase in the rate of rehydration in the early stage of the process (Rahman, 2015). The calculation of rehydration capacity can be made as follows (Ostermeier et al., 2020):

$$RC = \frac{M_r - M_d}{M_f - M_d} x100 \quad \text{(Eq. 3)}$$

where M_r is the mass of the rehydrated sample (g), M_d is the mass of the dried samples and M_f the mass of the fresh samples (g).

Rehydration has also been used as a quality parameter of dried pieces of fruits and vegetables. For example, one of these reports showed that freeze-drying was the best method for rehydration of kiwifruit pieces, followed by hot air–microwave assisted drying, hot air drying and vacuum drying. A preserved porous structure as the one achieved in freeze-dried products is favorable for rehydration (Akar & Barutçu Mazı, 2019). The use of perforations in infrared dried potato slices increased rehydration capacity (Rojas et al., 2019).

The use of a rapid freezing pretreatment and pulsed electric fields (PEF) enhanced rehydration capacity of strawberries and peppers, which was attributed to the increased number of pores in the membrane produced by PEF (Fauster et al., 2020). For hot air-dried onions, a PEF pretreatment increased the rehydration capacity in 47% (Ostermeier et al., 2020).

Freeze-dried tomatoes did not reach the same moisture content of fresh tomatoes after rehydration, which confirms the irreversibility of the drying process. The temperature dependence of rehydration capacity was well described by the Peleg and the Weibull models (Lopez-Quiroga et al., 2020). The use of osmotic dehydration, ultrasound and coating with pectin and ascorbic acid was the best strategy for increasing the rehydration capacity of hot air-dried pineapples. The result was attributed to the formation of cavities, fractures and microchannels due to ultrasound application (Sakooei-Vayghan et al., 2020). On the other hand, Zhou et al. (2021) stated that cavitation and mechanical effects promoted by ultrasound jeopardize rehydration of scallion stalks due to the damage caused to the tissue.

A study on microwave vacuum drying of okra showed that the optimal drying conditions for a obtaining the highest rehydration was high microwave power (900 W), intermediate vacuum level (60 kPa) and intermediate loading density (4 kg m^{-2}) (Xu et al., 2020). The use of cold air drying combined with ultrasound was favorable to the rehydration of potatoes, while complementation of drying with far infrared drying destructed the microstructure of the samples, jeopardizing rehydration (Zhang et al., 2020). Ohmic heating pretreatment of hot air-dried pineapple cubes was effective for increasing the mass gain after rehydration, i.e., ohmic heating showed a positive effect on rehydration capacity (Kumar et al., 2021).

For apple slices, the higher the drying temperature, the lower the rehydration capacity (Ullah et al., 2021). Such result can be extended to other raw materials, since high drying temperatures destroy the original vegetables microstructure, which is unfavorable to rehydration. For example, jujube slices that were hot air dried at high temperature presented collapse of the capillaries and reduced water retention capacity. On the other hand, PPD and especially freeze-drying reduced the collapsing area of the capillaries, which resulted in better rehydration (Wang et al., 2021).

To sum up, milder methods such as freeze-drying and innovative methods that preserve the original microstructure of dried fruit and vegetables consequently are more interesting for obtaining a better rehydration capacity.

2.5. Shrinkage

Plant foods shrink during drying proportionally to the elimination of moisture from the tissue. When drying rates are low, the product shrinks and become denser. When drying rates are high, case hardening takes place which limits shrinkage to some extent (Ordóñez, 2005). Shrinkage of fruit and vegetable pieces after drying has been assessed during the last years. In this sense, the solid displacement method and the toluene displacement method can be used to calculate percentual shrinkage by the following equation:

$$S = \frac{V_0 - V_f}{V_0} x100 \quad \text{(eq. 4)}$$

where S is shrinkage, V_0 is the sample volume before drying and V_f is the sample volume after drying.

Jerusalem artichoke pretreated by modified-cassava starch and sodium caseinate coating and dried by hot air drying and freeze-drying presented less shrinkage than control samples, without any pretreatment (Chottanom et al., 2020). The combination of image analysis and a vernier caliper was used for measuring shrinkage of strawberries and red bell peppers (Fauster et al., 2020). Results showed that the use of pulsed electric fields prior to freeze-drying reduced shrinkage, which was attributed to pore formation. On the other hand, for onions, the use of pulsed electric fields prior to hot air drying was found to increase shrinkage (Ostermeier et al., 2020). Osmotic dehydration and ultrasound-assisted osmotic dehydration reduced shrinkage in pectin-acid coated, hot air dried pineapples, which was attributed to the uptake of solids from the osmotic solution by the fruits and to the microcavities created by ultrasound, respectively (Sakooei-Vayghan et al., 2020).

Summarizing, usually, the use of pretreatments like coatings, pulsed electric fields, osmotic dehydration and ultrasound lead to decrease in shrinkage of dried fruit and vegetables, independent of the drying method used, which results in a better product quality.

2.6. Instrumental texture measurement

Food texture reflects human sensory perception when acting on food, mainly in the form of biting, yawing, grinding, etc., destroying or changing its structure and overall shape, making it suitable to be transferred to the stomach (Lu & Cen, 2013). Instrumental texture measurements are made by means of a texture analyzer. The most common parameter used for expressing dried fruit and vegetables texture is hardness (Aamir & Boonsupthip, 2017; Chottanom et al., 2020; Fauster et al., 2020; Hernández et al., 2020; Kowalska, Marzec, Domian, Masiarz, et al., 2020; Peng et al., 2018a, 2018b; Ullah et al., 2021; Wang et al., 2021).

Okra cubes dried by microwave presented a sharp increase in hardness during the first 1.5 min of drying followed by a sharp decrease until 5 min of drying and then stabilization until 7 min of drying. The hypotheses for explaining these results are crust formation followed by cooking (Aamir & Boonsupthip, 2017). The solid gain during osmotic dehydration increased hardness of carrot chips dried by DIC (Peng, Bi, et al., 2018). When subjected to freezing pretreatments, carrot chips dried by DIC presented lower hardness and higher crispness if frozen at −18, −40 and −80 °C (Peng, Yi, et al., 2018). Jerusalem artichoke slices presented higher hardness when dried by hot air drying as compared to freeze-drying (Chottanom et al., 2020). Firmness of strawberries and red bell peppers decreased with the application of PEF, a behavior that was attributed to the creation of pores due to PEF (Fauster et al., 2020). Both hot air drying and refractory window drying reduced the firmness of apple slices. Furthermore, osmotic pretreatment coupled with moderate electric field was not indicated as a pretreatment for refractory window drying of apples because it does not affect firmness at all (Hernández et al., 2020). The use of osmotic pretreatment combined with convection-microwave-vacuum drying led to higher breaking force and higher work in Japanese quince dried slices when compared to convective drying and freeze-drying (Kowalska, Marzec, Domian, Masiarz, et al., 2020). Bananas dried in a hybrid-solar-vacuum dryer presented a crispy texture when one vacuum pulse was used, which was confirmed by force-deformation curves with more significant irregularities (Roratto et al., 2021).

To sum up, results suggest that dried fruit and vegetables with crispy textures are well accepted by consumers. Pretreatments that improve crispness, that is related to higher porosity, include freezing, pulsed electric fields and osmotic dehydration. Innovative drying methods that improve crispness include convection-microwave-vacuum drying and hybrid-solar-vacuum drying.

2.7. Water activity

The water activity (a_w) of a food is the ratio between the vapor pressure of the food itself, when in a completely undisturbed balance with the surrounding air media, and the vapor pressure of distilled water under identical conditions (FDA, 2014). In drying studies, it is very interesting to measure water activity as it provides information on dried product shelf-life. Link et al. (2018) found that mango slices dried by conductive multi-flash drying with 4 cycles followed by vacuum drying (KMFD-VD) achieved very low values of water activity (0.226). Vacuum impregnation pretreatment accelerates the reduction of water activity in dried chestnuts (Yang et al., 2019). Osmotic pretreatment with sucrose solution coupled with moderate electric field led to a decrease in drying time needed to achieve an a_w of 0.4, which was attributed to electroporation (Hernández et al., 2020). Combined ultrasound-assisted osmotic dehydration (UOD) pretreatment at 35 kHz caused a higher decrease in water activity of dried apricots when compared to osmotic dehydration, which was attributed to an increase in the mass fraction of soluble solids (Sakooei-Vayghan et al., 2020).

In sum, the use of technologies like vacuum, osmotic pretreatment, electric field, and ultrasound lead to lower values of water activity in dried fruit and vegetable pieces.

3. Chemical analyses

3.1. Antioxidant activity

One of the greatest challenges facing food science and medical science is to effectively combat oxidation in food and human cells. Oxidative stress is a pathological condition in which reactive oxygen species/nitrogen species (ROS/RNS) modify biological macromolecules, causing tissue damage and accelerating cell death in the body (Apak et al., 2016) and nutritional loss in food matrices. Although, there is no single method to evaluate the antioxidant potential, thus usually two or more methods are combined in each study. The most common methods used in food studies are 2,2-Diphenyl-1-picrylhydrazyl - DPPH, [2, 2′-Azinobis-(3-Ethylbenzthiazolin-6-Sulfonic Acid)] - ABTS (Mixed-Mode methods; electron transfer ET + hydrogen atom transfer HAT), Fluorescence recovery after photobleaching - FRAP, Cupric Ion Reducing Antioxidant Capacity - CUPRAC and Oxygen radical absorbance capacity - ORAC (ET methods).

The meaning of antioxidant essays in drying studies is to verify the effect of temperature, drying methods and treatments in the reduction of antioxidant activity. Lopez et al. (2017) stated that time was more significant than temperature when studying the vacuum drying murta berries, as the temperature 50 °C got lower values of antioxidant activity than 90 °C, as the process was longer. In general, DPPH and ORAC methods presented around 30% loss of antioxidant activity at the optimal temperature. Drying is one of the most harmful procedures to bioactive compounds, due to the degradation of biologically active compounds at high temperatures from chemical, enzymatic, or thermal decomposition (Lopez et al., 2017).

Nehra and Deen (2017) concluded that Kankoda fruits lost 10–20% of antioxidant activity after 4 days of oven-drying at 55 °C. Thus, even the considered low-temperature drying promotes losses of bioactive compounds. For beetroots drying, Nistor et al. (2017) found a combination of answers with DPPH essay: higher temperature decreases antioxidant activity, and a combination of methods, as microwave and forced convection drying, reduces drying time and improve by 3 times antioxidant activity, when compared to free convection. Therefore, antioxidant essays can help with several analyses of parameters inside a drying optimization.

Filiz and Seydim (2018) produced hot air dried apple chips and assessed several quality parameters, including ORAC and Trolox equivalent antioxidant capacity - TEAC. They found that antioxidant activity in apple chips increased when compared to raw apples, especially for higher hot air drying temperatures (75 °C). The authors hypothesized that bound phenolics may have been released from the breakdown of cell constituents, which occurred by the effect of drying heat, thus increasing antioxidant activity.

Comparison of methods is one of the most common drying studies, alongside the evaluation of time and temperature parameters' optimization. In Sehrawat et al. (2019) investigation, red onion, Indian gooseberry, papaya, carrot and sweet potato showed that vacuum drying gets higher antioxidant activity (by DPPH) than hot air drying. Moreover, not just treatments are evaluated, pre-treatments can be also investigated by antioxidants results. For example Vallespir et al. (2019) stated that freezing treatments, before convective drying, may help with microstructure although decrease significantly antioxidant activity (FRAP, CUPRAC, ABTS) for the following food items: beetroots, apple slices and eggplant. Hence, usually antioxidant essays demonstrate how negatively the drying process affect food matrices.

The effect of edible coating on antioxidant activity and physical properties of dried Jerusalem artichoke slices was studied by Chottanom et al. (2020), who found that the antioxidant activity of coated samples was higher than that of uncoated ones. When comparing sodium caseinate and cassava modified starch as coating materials, the former showed higher DPPH radical inhibitory activity. Such result was attributed to sodium caseinate oxygen-proof properties. Another technique used for increasing antioxidant activity of dried fruit pieces is osmotic dehydration, which presents novel features such as the use of fruit juices as osmotic solutions. In this sense, Kowalska, Marzec, Domian, Galus, et al. (2020) used chokeberry juice as osmotic solution for treating apples prior to drying by freeze-drying and hot air/-microwave vacuum drying (hybrid method). They discovered that no difference between drying methods was found with regard to DPPH antioxidant activity, while the use of chokeberry juice improved it significantly when compared to sucrose. Such difference was attributed to the presence of phenolic compounds in chokeberry juice.

The use of modern solar dryers can be an alternative to save energy and increase antioxidant activity in fruit and vegetable pieces. In the report of Al Maiman et al. (2021), tomato slices were solar dried and had their quality assessed by means of several ways, including DPPH radical scavenging activity. Results showed that right after drying, there was no significant increase in antioxidant activity. However, storing solar dried tomato slices for 90 and 180 days significantly increased their DPPH radical scavenging activity, a result that was attributed to the increased release of phytochemicals from the vegetable matrix.

Summarizing, drying time seems to be a key-process parameter affecting antioxidant capacity of dried fruit and vegetable pieces, though the effect of drying temperature cannot be neglected.

3.2. Total phenolics

Usually, research papers relate total phenolic content with antioxidant activity, as phenolic compounds act as natural antioxidants, and results are often expressed in mg of GAE (gallic acid equivalent, used as standard) per g or 100 g (d.m. – dry matter). López et al. (2017) corroborated antioxidant activity, stating that 50 °C drying temperature and its longer associated time is more harmful to phenolics than 60 and 70 °C. As well as Nistor et al. (2017), in which total phenolics found in the combination of methods, namely microwave and forced convection drying, resulted in higher results by 2–3 times.

As discussed for antioxidant capacity, Filiz and Seydim (2018) observed that drying increased phenolic content of apple slices. Such effect was more intense for higher drying temperatures. It was attributed to the release of bound phenolics from the tissue due to hot air heat.

Kondareddy et al. (2019) found that solar and open-sun dried turmeric slices presented 54.8% and 63.8% less total phenolic compounds when compared to fresh turmeric. Sometimes, pretreatments are used in an attempt to increase the quality of dried fruit and vegetable pieces, but the objective is not completely achieved. For example, Vallespir et al. (2019) used freezing before hot air drying of beetroot, apple and eggplant, finding that such pretreatment increased the losses of phenolic compounds caused by drying.

In Sehrawat et al. (2019) results, red onion, and papaya showed a decay in phenolic content with drying up to 50%, while the Indian gooseberry, carrot and sweet potato samples, which have naturally less water in its composition, got up to 30% phenolic loss. Regarding the treatments, vacuum drying retained more phenolic compounds than hot air drying. Similarly to Sehrawat et al. (2019), Bozkir (2020) tested two vacuum dryers against hot air drying and stated the superiority of the vacuum options, this time for orange slices. Nevertheless, Bozkir (2020) did not use antioxidant activity as confirmation as Kowalska, Marzec, Domian, Galus, et al. (2020) study in apple slices. Phenolic content confirmed the significant improvement no matter the drying method (puffing or freeze-drying) applying a type of osmotic solution so called "sucrose with concentrated chokeberry juice". This highlights the importance of not focusing only on the drying method, but also on pre-treatments to protect bioactive compounds. Bozkir (2020) and Kowalska, Marzec, Domian, Galus, et al. (2020) used the vitamin C analysis to confirm phenolic results.

As discussed for antioxidant capacity, innovative osmotic solutions can also be used for increasing the phenolic content of dried fruit and vegetable pieces. In the report of Maleki et al. (2020), they found that the use of a roselle extract rich in polyphenolic compounds as part of the solution for osmotic impregnation-dehydration increased the phenolic content of carrots. Subsequently, carrots hot air dried at 70 °C presented the highest phenolic content. Such result was attributed to faster drying, when compared to a drying temperature of 60 °C, combined with mild drying temperature, when compared to a drying temperature of 80 °C.

Innovative drying methods also yield promising results when it comes to phenolic compounds preservation. In this sense, Xu et al. (2020) discovered that microwave vacuum dried okra presented similar phenolic content to freeze-dried samples and both were superior to hot air dried samples. In this case, the mechanism responsible for phenolics destruction during hot air drying was claimed to be high temperature. In addition, Zhang et al. (2020) used ultrasound-strengthened cold air drying combined with sequential far-infrared radiation drying (UCAD-FIRD) to process potato slices, finding that such innovative method was fundamental for a better preservation of phenolic compounds in the samples when compared to cold air drying and ultrasound-strengthened cold air drying. The increase in temperature due to radiation was claimed to inhibit phenolic compounds oxidation. Furthermore, Al Maiman et al. (2021), using an innovative solar dryer, found that total phenolic content of tomato slices was completely preserved after drying on day 0 of storage and increased after 90 and 180 days.

To sum up, such results suggest that low drying time, vacuum methods and the use of pretreatments preserve more phenolic compounds, which is possibly related to the protection against oxidative reactions achieved when using such conditions and techniques.

3.3. Vitamin C

Vitamin C analysis can be used as an antioxidant method to confirm phenolic content, normally expressed in terms of ascorbic acid (mg/100 g d.m.). As did Bozkir (2020) which found more vitamin C content in orange slices dried by two vacuum dryers (microwave and infrared) than the one dried with hot air drying. Similarly, Kowalska, Marzec, Domian, Galus, et al. (2020), when working with dried apple slices, confirmed the most efficient pre-treatment to be the application of an osmotic solution, namely sucrose with concentrated chokeberry juice, resulting in 23–32% more ascorbic acid content than the fresh sample.

Conversely, sometimes an increase in temperature helps to preserve vitamin C, such as in the study of Zhang et al. (2020). Possibly, the use of ultrasound-strengthened cold air drying combined with sequential far-infrared radiation drying (UCAD-FIRD) reduced the drying time of potato slices and the exposition of their vitamin C to oxidation.

Vitamin C is an expression of fruit quality and its loss is attributed to long-term exposure to high temperatures and long storage times (Wang et al., 2021). With another perspective, Al Maiman et al. (2021) used vitamin C to state the stability of solar dried tomato slices during shelf-life. As this compound is known to decay rapidly in processed food, the results of Al Maiman and collaborators were promising, as to invest more in solar technology, since vitamin showed great stability during 180 days.

Regarding the evaluation of methods, vitamin C content may also help with optimization. Wang et al. (2021) found that, for jujube crisp slices, hot air drying (80 °C–15 h) is highly more harmful to vitamin C than freezing and puffing drying, which confirms the negative effect of high temperatures.

Summarizing, given the high susceptibility of vitamin C to oxidation, it was expected that vacuum methods would preserve better this compound than traditional methods. Surprisingly, solar drying showed high potential of preservation of vitamin C as well. Long drying times and high drying temperatures are also known for destroying vitamin C, and for dried fruits and vegetablesthis was also observed.

4. Other analyses

4.1. Microbiological counts

This analysis usually reflects the dried food safety for consumptions and/or conditions of hygiene during preparation. Several researchers have been assessing the microbiological quality of their dried products. Ultrasound-assisted osmotic dehydration of apricots followed by coating with pectin/citric or ascorbic acid and hot air drying led to decrease in water activity, and consequently, to decrease in total mesophilic aerobic count and total yeast and mold count (Sakooei-Vayghan et al., 2020). Another report evaluated the shelf-life of osmodehydrated cabbage as packaged in different modified atmospheres and stored under refrigeration (Cvetkovic et al., 2021). Results showed that in the beginning of storage, the total number of microorganisms decreased due to the absence of oxygen in the modified atmosphere packaging. In addition, at the end of storage, the number of microorganisms increased, probably due to anaerobic microorganisms' growth. In some cases, microbiological tests are made with the aim of quantifying probiotic bacteria added to dried products (Li et al., 2021). Solar-drying was found to reduce the average microbial population in tomato slices when compared to fresh samples (Al Maiman et al., 2021). Scallion stalks pretreated by ethanol or ethanol and ultrasound and subsequently dried by infrared convection drying presented microbial quality within the limits required by the Chinese National Standard (Zhou et al., 2021).

To sum up, usually, drying reduces water activity and yields products with long shelf-life. Osmotic-dehydration, when used as a pretreatment for drying, also produces shelf-stable products, but when used alone does not assure shelf-stability. Water activity values below 0.6, which is usually achieved in drying of fruit and vegetable pieces, inhibit microorganisms' growth.

4.2. Sensory analysis

Product quality measurement by human subjects is the purpose of sensory analysis, which can provide data such as overall acceptability, flavor, aroma, texture, color, and appearance. Sensory evaluation can be divided into difference tests, affective (consumer) tests, and descriptive analysis (trained panels) (Duizer & Walker, 2016). Both can access which treatment was better to dry certain fruit or vegetable, as sensory quality affects directly the purchase intention.

Microwave dried okra presented better color, texture, flavor, and overall acceptability when an 800 W microwave power and a 4 min drying time were used (Aamir & Boonsupthip, 2017). Dried carrots obtained by osmotic dehydration/Instant Controlled Pressure Drop Drying presented better sensory quality when maltitol solutions at 60° Brix concentration were used for osmotic dehydration for 6 min (Peng, Bi, et al., 2018). Apple slices osmotically dehydrated in chokeberry juice and dried by freeze drying or convection-microwave-vacuum drying presented low sensory scores, which was attributed to sour, tart taste and too dark red color (Kowalska, Marzec, Domian, Galus, et al., 2020). Infrared-assisted spouted bed drying at 40 °C and 22 m/s was the best condition for producing yam snacks with best sensory quality (Li et al., 2021).

In sum, sensory quality is a key-feature of any food, as it controls its purchase intention. For obtaining dried fruits and vegetableswith high sensory quality, it is necessary to test several process conditions and performing sensory tests. Usually, hedonic tests are used for this purpose. There is not a general rule in this case: the optimal conditions must be found by using sensory tests.

4.3. Overview of drying methods used for fruits and vegetables

Over the last five years, several drying methods have been used in the studies dedicated to dried fruits and vegetables. This section is devoted to present the features of these methods, along with their advantages

Table 1
Studies conducted over the last 5 years on quality assessment of dried fruits and vegetables obtained by hot air drying: quality analyses performed, drying method advantages and disadvantages.

Reference	Product	Method	Quality Analyses	Advantages	Disadvantages
Filiz and Seydim (2018)	Apple slices	Hot air drying (HAD)	Chemical: ascorbic acid, total phenolics, total flavonoids, antioxidant activity (ORAC[a, b, c, d, e and f], TEAC[b]), hydroxymethyl furfural (HMF), phenolics (HPLC[c])	Increased phenolics, flavonoids and HMF content, increase in antioxidant activity	Ascorbic acid degradation
Vallespir et al. (2018)	Beetroot/apple/eggplant cubes	HAD (freezing pretreatment)	Physical: microstructure, color	Increased drying rates	Noticeable color changes, contraction, and collapse
Zhang et al. (2018)	Carrot/sweet potato/yellow bell pepper/broccoli slices	HAD	Physical: microstructure Chemical: carotenoids (HPLC), cell wall analysis	Preserved α-carotene in all assessed vegetables, increased carotenoid bioaccessibility in carrot and yellow bel pepper	High degradation of β-carotene in carrot, sweet potato, and yellow bell pepper; decreased carotenoid bioaccessibility in sweet potato and broccoli
Vallespir et al. (2019)	Beetroot/apple/eggplant cubes	HAD (freezing pretreatment)	Physical: microstructure, texture Chemical: total phenolics, antioxidant activity (FRAP[d], CUPRAC[e], ABTS[f])	Increased drying rates	Texture alteration, phenolics and ascorbic acid degradation
Ge et al. (2020)	Peppers	HAD	Chemical: volatile flavor compounds	High flavor retention at 60 °C	High volatiles loss at 70–80 °C
Obajemihi et al. (2020)	Tomato slices	HAD	Physical: color	Red hue	High ΔE
Ostermeier et al. (2020)	Onion discs	HAD (PEF pretreatment)	Physical: color, blisters, shrinkage, rehydration capacity Chemical: pyruvic acid	Shortened drying time, low ΔE, absence of blisters, high rehydration capacity, high pyruvic acid content	Shrinkage
Kumar et al. (2021)	Pineapple cubes	HAD (ohmic pretreatment)	Physical: rehydration capacity, color, texture, total soluble solids (TSS)	Low ΔE, high rehydration capacity, acceptable textural degradation	Decreased TSS (leaching during pretreatment)
Nwakuba et al. (2021)	Garden egg, cucumber, and white carrot slices	HAD	Physical: shrinkage	Low shrinkage for cylindrical-shaped garden egg sample	High shrinkage for spherical-shaped cucumber sample
Oshima et al. (2021)	Persimmon cylinders	HAD (blanching pretreatment)	Physical: color, texture, nanostructure Chemical: galacturonic acid	Shortened drying time, intensified color, hardness decrease	Flow of carotenoids into the blanching water, nonenzymatic browning
Ullah et al. (2021)	Apple slices	HAD	Physical: texture, rehydration capacity	High rehydration capacity, low moisture content (for optimized conditions)	Low drying rates (for optimized conditions)

[a] Oxygen radical absorbance capacity.
[b] Trolox equivalent antioxidant capacity.
[c] High performance liquid chromatography.
[d] Fluorescence recovery after photobleaching.
[e] Cupric Ion Reducing Antioxidant Capacity.
[f] [2,2′-Azinobis-(3-Ethylbenzthiazolin-6-Sulfonic Acid)].

and disadvantages for the processing of dried fruits and vegetables.

The simplest drying method available for drying fruits and vegetables regarding the energy source is solar drying, which consists in using solar dryers. The mechanism of solar drying is directly related to the construction of the solar dryers. In this sense, one of the key elements of a solar dryer is the solar collector, which is the primary energy source for a solar dryer. Basically, it converts and transfers energy. In this sense, the direct and diffuse radiation coming from the sun is converted by the absorber of the collector absorber into heat. Then, heat is transferred to the working medium of the collector, which can be the drying air, in the case of direct solar dryers or a liquid, in the case of indirect solar dryers (Mujumdar, 2015).

Probably the most popular drying method for fruits and vegetables is convection drying. In such case, the heat source is heated air or gas flowing over the solid surface. Evaporation is possible due to the heat supplied by convection to the solid surface. The evaporated moisture is carried away by the drying medium, which can be air (most common), inert gas (such as N_2), direct combustion gases, or superheated steam (Mujumdar, 2015).

Another technique used for drying fruits and vegetables is conventional vacuum drying. It occurs in the absence of oxygen and at mild temperature, which results in the preservation of most of the food nutrients and sensory quality. During vacuum drying, pressures under the atmospheric are achieved inside the drying chamber. Therefore, it is possible to evaporate the water contained in the food at lower temperatures when compared to atmospheric drying (Richter Reis, 2014). For heat-sensitive fruits and vegetables, the vacuum drying process of choice is freeze-drying. In this process, initially, the food is frozen solid; then it is exposed to a controlled temperature-pressure environment in a chamber; the pressure in the chamber is regulated as to promote sublimation of solid ice, which takes place when pressure is reduced below 0.006 atm (Maguire, 1967). For fruits and vegetables that must be dried fast, the vacuum drying process of choice is microwave-vacuum drying. While a typical convective drying usually takes hours, a typical microwave-vacuum drying takes minutes. The use of microwaves in drying improves the drying rates due to the porous structure created during water evaporation by dielectric energy, facilitating the mass transfer (Drouzas et al., 1999). In a microwave-vacuum dryer, heat is obtained by electromagnetic radiation. Such energy is absorbed by the water molecules contained in food and converted into kinetic energy (vibration), which generates heat that is used for their evaporation. Such process, combined with vacuum, results in high drying rates at low temperatures (Mousa & Farid, 2002). Microwave drying of fruits and vegetables can also be carried out at atmospheric pressure. In this case, care must be taken not to burn the product, since hot spots can occur in microwave heating.

Infrared drying, either under vacuum or combined with different types of convective drying, has been used for drying fruits and

Table 2
Comparative studies conducted over the last 5 years on quality assessment of dried fruits and vegetables obtained by various conventional drying methods: quality analyses performed, drying methods' advantages and disadvantages.

Reference	Product	Methods	Quality Analyses	Advantages	Disadvantages
Farhaninejad et al. (2017)	Banana slices	1 Osmotic dehydration (OD) using direct sonication (US) 2 OD using indirect US	Physical: total color difference, surface area change	1 increased water loss 2: reduced solid gain	1 increased solid gain 1 and 2 increased total color difference and surface area change
Nistor et al. (2017)	Beetroot slices	1 Hot air drying (HAD) (free convection) 2 HAD(free convection) followed by HAD (forced convection) followed by microwave (MW)	Physical: microstructure Chemical: betalain, betacyanin, betaxanthin, antioxidant activity (DPPH) and total phenolics	2 increased betalain extraction, high betacyanin, betaxanthin and phenolic contents and antioxidant activity	1 long drying time, tissue shrinkage and collapse
Smith et al. (2018)	Tomato slices, mango slices	1 Solar drying (SD) 2 Ceramic zeolite drying beads	Chemical: β-carotene, α-tocopherol, ascorbic acid	1 and 2 preservation of ascorbic acid and α-tocopherol in mango 2 preservation of ascorbic acid in tomato	1 decrease in ascorbic acid in tomato 1 and 2 degradation of β-carotene in mango; degradation of α-tocopherol and β-carotene in tomato
Akar and Mazi (2019)	Kiwi slices	1 HAD 2 Vacuum drying (VD) 3 Freeze-drying (FD) 4 Microwave vacuum drying (MWVD)	Physical: rehydration capacity, color Chemical: ascorbic acid	3 preserved color, high bioactive retention, high water absorption rate and shape retention 4 short drying time, high overall quality	3 long drying time
Kondareddy et al. (2019)	Black turmeric slices	1 SD 2 Sun drying	Physical: color Chemical: total phenolics, total flavonoids, antioxidant activity (DPPH[a])	1 high antioxidant activity preservation	2 high bioactive degradation
Sehrawat et al. (2019)	Onion shreds, Indian gooseberry slices, semi-ripened papaya slices, carrot shreds, sweet potato slices	1 VD 2 HAD	Physical: a_w, color Chemical: total antioxidant capacity, total phenolics	1 high color retention 1 and 2 low a_w	1 and 2 decrease in antioxidant capacity and phenolic content
Yang et al. (2019)	Chestnut slices	1 VD 2 FD 3 HAD (vacuum impregnation pretreatment)	Physical: soluble solids, a_w, microstructure, color Chemical: Iron Chemical: acidity, total phenolics, vitamin C	2 preserved color and microstructure preservation	3 high browning
Chottanom et al. (2020)	Jerusalem artichoke slices	1 HAD 2 FD(coating pretreatment)	Physical: texture, color shrinkage Chemical: antioxidant activity (DPPH) Other: sensory analysis	2 low ΔE, low browning index, low shrinkage	1 high shrinkage
Kowalska, Marzec, Domian, Masiarz, et al. (2020)	Japanese Quince slices	1 OD* followed by HAD 2 OD followed by FD 3 OD followed by HAD followed by MWVD *chokeberry/apple juice	Physical: texture, a_w, color Other: sensory analysis	1 and 2 high crispness 2 low ΔE 2 and 3 high sensory scores for color	1 visible browning 3 excessive hardness
Kowalska, Marzec, Domian, Galus, et al. (2020)	Apple slices	1 OD* followed by FD 2 OD followed by HAD followed by MWVD *chokeberry juice	Chemical: vitamin C, total phenolics, antioxidant activity (DPPH) Other: sensory analysis	1 and 2 increase in vitamin C, phenolics, and antioxidant activity	1 and 2 low sensory scores
Xu et al. (2020)	Okra pieces	1 MWVD 2 HAD 3 FD	Physical: color, rehydration capacity Chemical: total phenolics, flavonoids	1 high phenolic and flavonoid content 1 and 3 intense green color, high rehydration capacity	2 red color, low rehydration capacity

[a] 2,2-Diphenyl-1-picrylhydrazyl.

vegetables (Bozkir, 2020; Li et al., 2021; Rojas et al., 2019; Zhang et al., 2020; Zhou et al., 2021). Infrared radiation comprises electromagnetic waves of wavelength between 0.78 and 1000 μm, which are used to provide heat to the food, thus allowing water evaporation during the infrared drying process.

Osmotic dehydration can be considered a pretreatment to drying and a processing technique as well, since it yields intermediate moisture foods like dried fruits and vegetables. Such process consists in soaking

Table 3
Studies conducted over the last 5 years on quality assessment of dried fruits and vegetables obtained by innovative drying methods: quality analyses performed, drying methods' advantages and disadvantages.

Reference	Product	Methods	Quality Analyses	Advantages	Disadvantages
Cao et al. (2018)	Winter jujube slices	Pulsed Air-Jet Impingement Drying	Physical: color	Preserved color at low drying temperatures (~55 °C)	Browning at high drying temperatures (~75 °C)
Link et al. (2018)	Mango slices	1 Conductive multi-flash drying (KMFD) 2 KMFD followed by vacuum drying (VD)	Physical: a_w, true volume, bulk volume, bulk density, accessible porosity, color, microstructure, texture	1 and 2 Fast drying, high porosity, high L*, low ΔE, crispy or soft texture 2 reduced number of heating-vacuum pulses	1 high number of heating-vacuum pulses
Peng, Bi, et al. (2018)	Carrot cuboids	Osmotic dehydration (OD) followed by Instant Controlled Pressure Drop Drying (DIC)	Physical: volume, texture, microstructure, color Other: sensory analysis	Highly porous structure, high sensory acceptability, crispness, acceptable color	Poor sensory quality for OD at 20°Brix for 15 min
Peng, Yi, et al. (2018)	Carrot cuboids	OD followed by DIC (freezing pretreatment)	Physical: volume, texture, color, microstructure, and pore size distribution	Low ΔE, low hardness, high crispness, homogeneous porous structure	Severe shrinkage and excessive hardness for N_2 frozen samples
Rojas et al. (2019)	Potato slices	Infrared drying (perforation and ethanol pretreatments)	Physical: rehydration capacity	Short drying time, improved rehydration capacity	Crust formation
Bozkir (2020)	Orange slices	1 Microwave vacuum drying (MWVD) 2 Vacuum infrared drying 3 Hot air drying (HAD)	Physical: pH, bulk density, color Chemical: acidity, total phenolics, vitamin C	1 High drying rates, high effective water diffusivity, high product overall quality	3 low product overall quality
Hernández et al. (2020)	Apple slices	1 OD followed by refractance window 2 ODfollowed by HAD	Physical: texture, a_w, soluble solids, color, microstructure	1 short drying time, highly porous structure	1 brown color of samples dried at 75–95 °C
Zhang et al. (2020)	Potato slices	1 Ultrasound (US) assisted cold air drying 2 US assisted cold air drying followed by far-infrared radiation drying	Physical: color, browning, rehydration capacity Chemical: total phenolics, vitamin C	1 and 2 high bioactive preservation, color quality and rehydration capacity 2 shortened drying time (as compared to 1)	1 long drying time
Li et al. (2021)	Yam cubes	Infrared-assisted spouted bed drying	Physical: color, microstructure Chemical: polysaccharide yield Other: sensory analysis, microbial counts, storage stability	Probiotic preservation, high polysaccharide content, high whiteness index, acceptable sensory quality, long shelf-life	Long drying time
Roratto et al. (2021)	Banana, persimmon, and carrot slices	Hybrid solar vacuum drying (KMFD)	Physical: porosity, a_w, microstructure, texture, porosity, bulk density	Large and well-distributed pores, crispy texture, high porosity, low bulk density	High number of heating-vacuum pulses at specific conditions
Wang et al. (2021)	Jujube slices	1 HAD 2 Freeze-drying (FD) 3 Pressure-differential puffing drying (PPD)	Physical: color, apparent density, rehydration capacity, texture, microstructure Chemical: vitamin C Other: biological availability	2 high color quality, low apparent density and shrinkage, high rehydration capacity, and vitamin C content 3 high bioavailability, preserved microstructure	1 excessive hardness, high apparent density, low rehydration capacity, low vitamin C content, severe shrinkage, browning
Zhou et al. (2021)	Scallion stalks slices	Infrared convection drying (US and ethanol pretreatments)	Physical: rehydration capacity, color, microstructure, water distribution Chemical: volatile compounds Other: microbial counts	Shortened drying time, increased rehydration capacity, improved color and flavor, low microbial counts	Damaged cell structure, loss of volatile components

food pieces in a concentrated solution containing a suitable humectant (usually sugar in the case of fruits or salt in the case of vegetables). Osmotic pressure causes water to diffuse from the food into the solution. The humectant replaces the lost water (Fellows, 2017).

Innovative drying methods have been used in the investigations dedicated to dried fruits and vegetables too. One example is instant controlled pressure drop (DIC) drying, which consists of, initially, a quick heating step (10–60 s) including a saturated steam injection at high-pressure (up to 1 MPa) applied to product, which is placed initially under vacuum. In this step, vapor condensation and product heating take place, and the product moisture content increases by 0.1 g H_2O/g dry basis. Sometimes, compressed air could be used as a pressurized agent. Then, an abrupt dropping of pressure (0.5 MPa s^{-1}) toward a vacuum (3–5 kPa) over only 10–60 ms results in evaporation of water within the product, which produces vapor evaporation, cooling, and volume expansion (Hamoud-Agha & Allaf, 2020). Another innovative drying process is conductive multi-flash drying (KMFD). In multi-flash drying processes, the product is heated at atmospheric pressure until the desired temperature, before the application of a vacuum pulse that leads to flash evaporation, which in turn causes the product cooling. At the end of the process, various heating vacuum pulses cycles are applied, yet most recent studies on this theme have tested a lower number of heating vacuum pulses cycles with very good results. The heating medium can be either hot air or hot plates. In the latter case, i.e., when the drying energy is supplied by heat conduction, the process is called conductive multi-flash drying (KMFD) (Link et al., 2018).

Tables 1–4 show the reports published over the last 5 years on quality assessment of dried fruits and vegetables obtained by hot air drying, various conventional drying methods (comparative studies), innovative drying methods and other drying methods, respectively. The tables bring the drying methods used, the quality analyses that have been carried out and the advantages and disadvantages of using such drying methods for the process and for the dried product quality. It can be realized from the tables that hot air drying is the most popular drying method for fruits and vegetables. Additionally, studies comparing different drying methods are also very usual. Finally, the use of more than one drying

Table 4
Studies conducted over the last 5 years on quality assessment of dried fruits and vegetables obtained by other drying methods: quality analyses performed, drying methods' advantages and disadvantages.

Reference	Product	Method	Quality Analyses	Advantages	Disadvantages
Aamir and Boonsupthip (2017)	Okra cubes	Microwave (MW)	Physical: color, texture Other: sensory analysis	Short drying time, high greenness preservation, hardness similar to fresh product, high sensory acceptability	Product browning
López et al. (2017)	Murta berries	Vacuum drying (VD)	Chemical: proximate composition, free and bound total phenolics and total flavonoids, phenolics, flavonoids, sugars, and β-carotene (HPLC[a]), and antioxidant activity (DPPH[b], ORAC[c])	Increased β-carotene content	Decreased phenolic content and antioxidant activity
Nehra and Deen (2017)	*M. dioica* slices	Shade drying followed by hot air drying (HAD)	Chemical: total phenolics, total flavonoids, ascorbic acid, antioxidant activity (DPPH, FTC[d]), β-carotene	Shelf-stable product presenting radical scavenging capacity	Decreased phenolic and flavonoid content
Bora et al. (2018)	Apple slices	VD	Physical: color	Acceptable color changes for most apple varieties at 70–80 °C	Severe color changes at 60 °C and at 80 °C for one apple variety
Fauster et al. (2020)	Strawberries/red bell pepper cylinders	FD (pulsed electric field - PEF pretreatment)	Physical: shrinkage, rehydration capacity, texture	Decreased shrinkage and firmness, increased rehydration capacity	Long drying time
González-Toxqui et al. (2020)	Grapes	FD (alkaline emulsion pretreatment)	Physical: microstructure, rehydration capacity Other: microbial load	Decreased drying time, energy saving, reduced microbial load	Loss of sensory quality in the rehydrated product
Li et al. (2020)	Apple cubes	HAD followed by moisture equilibrium process (MEP) followed by DIC	Physical: water distribution, volume expansion ratio, microstructure, porosity, pore size distribution	Absence of shrinkage, light color, uniform water distribution, expanded volume, fully expanded pores, smooth surface, high porosity, broad pore size distribution	Long processing time
Lopez-Quiroga et al. (2020)	Tomato pieces	FD	Physical: a_w, rehydration capacity, microstructure	Highly interconnected porous microstructure, fast rehydration	Long drying time
Maleki et al. (2020)	Carrot cylinders	OD* followed by HAD *roselle extract (US pretreatment)	Physical: microstructure Chemical: total phenolics	Increased nutritional value	Shrinkage
Sakooei-Vayghan et al. (2020)	Apricot cubes	OD followed by HAD (US pretreatment)	Physical: soluble solids, a_w, shrinkage, apparent density, bulk density, rehydration capacity, browning, microstructure Other: microbial counts	Decreased shrinkage, apparent density, bulk density, microbial count and a_w, increased rehydration capacity, and improved texture	Browning
Al Maiman et al. (2021)	Tomato slices	SD	Physical: a_w, browning, color Chemical: acidity, vitamin C, carotenoids, lycopene, total phenolics, total flavonoids, antioxidant activity (DPPH) Other: microbial counts	Decreased microbial counts, elimination of pathogenic bacteria; increased carotenoid, lycopene, phenolic, and flavonoid content, and antioxidant activity	Decreased vitamin C content, browning
Cvetkovic et al. (2021)	Cabbage slabs	OD (modified atmosphere packaging)	Physical: pH, a_w Chemical: acidity, L-ascorbic acid Other: microbial counts, sensory analysis	Decreased microbial counts, acceptable sensory quality	Decreased ascorbic acid content

[a] High performance liquid chromatography.
[b] 2,2-Diphenyl-1-picrylhydrazyl.
[c] Oxygen radical absorbance capacity.
[d] Ferric thiocyanate.

method in a sequential combination is a trend.

5. Conclusion

Dried fruits and vegetables are a very popular food product all around the world. Their quality assessment is a very important step for their commercialization. In this sense, many different quality analyses have been carried out in scientific reports over the five last years. In addition, many drying methods have been used in these investigations.

Results presented in this report show that dried fruits and vegetables with low enzymatic and non-enzymatic browning, low apparent density, preserved microstructure, high rehydration capacity, low shrinkage, crispy texture, and low water activity are considered as having a good physical quality. Additional results confirm that dried fruits and vegetables with high antioxidant activity, total phenolics and vitamin C are considered as having a high chemical/bioactive quality. Finally, dried fruits and vegetables must present low microbial counts and high sensory acceptability. Such physical, chemical, microbiological, and sensory quality can be achieved by using innovative pretreatments, mild drying conditions, mild drying methods and innovative drying methods.

In conclusion, the quality of dried fruits and vegetables is directly affected by the drying method chosen. Innovative pretreatments capable of changing the drying kinetics and the product quality seem to be a trend. With regard to drying, the use of infrared heating, solar energy and vacuum are promising technologies.

Declaration of competing interest

Felipe Richter Reis, Caroline Marques, Ana Carolina Sales de Moraes and Maria Lucia Masson declare that they have no conflict of interest.

Data availability

No data was used for the research described in the article.

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NOTES ON NEGLECTED AND UNDERUTILIZED CROPS

Sea fennel (*Crithmum maritimum* L.): from underutilized crop to new dried product for food use

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Abstract Sea fennel (*Crithmum maritimum* L.) is a perennial halophyte species typical of coastal ecosystems, used fresh in traditional cuisine and folk medicine due to its sensory properties and a good content of healthy compounds. Although considered as a promising biosaline crop, this halophyte is underutilized for commercial cultivation possibly due to a shortage of its consumer demand. For promoting a full exploitation of this species, a new food product was obtained by drying sea fennel using different treatments (air-drying, microwave-drying, microwave-assisted air-drying and freeze-drying). Water activity, essential oil content, chlorophylls, surface colour, colouring power and sensory evaluation were analyzed. All drying treatments allow to obtain a good water activity but significantly reduced the content of essential oils and chlorophylls. Freeze-drying and microwaving preserved the surface colour parameters more than other drying treatments, while freeze-drying gave the product the best colouring power. Based on sensory analysis, microwave-drying, microwave-assisted air-drying and freeze-drying showed the highest scores among the drying methods. Taken together the results indicate that microwaving and freeze-drying are optimal for preserving qualitative traits, including organoleptic properties, in dried sea fennel for food use. Furthermore, dried sea fennel can be usefully exploited in human food not only for its aromatic traits but also for its food colouring power like other plant derived natural colorants. It could be concluded that this underutilized crop could play a better role for making up a sustainable food production system.

Keywords Aromatic herb · Colouring power · *Crithmum maritimum* L. · Exploitation · Halophyte · Sensory evaluation

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Introduction

Sea fennel (*Crithmum maritimum* L.), the only species of the genus *Crithmum*, is an aromatic herb (Pistrick 2002) belonging to the Apiaceae family. Sea fennel is also known as crest marine, marine fennel, samphire and rock samphire because it grows wild on maritime rocks, breakwaters and sandy beaches (Fig. 1). In German it is named *Meerfenchel* or *Seefenchel*; in French *fenouil marin* or *passepierre*; in Italian *finocchio marino* or *critama*; in Turkish *kaya koruğu* or *deniz rezenesi* (Franke 1982; Özcan et al. 2001). The

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Fig. 1 Plant of sea fennel on a sandy beach as typical example of natural habitat for this halophyte

term *Crithmum* derived from Greek *krithe* (barley), probably due to the resemblance of fruits to barley-corns; *maritimum* is due to its habitat, the sea (Atia et al. 2011). *C. maritimum* is a highly branched plant of up to 30–60 cm high with a strong, thick and gnarled root. Leaves are succulent and have a sheath-like base with the short petiole ending in a pinnate composed blade, which is usually divided into three leaflets 2–5 cm long (Franke 1982). The plant blooms between June and September, while the fruit begins to ripen in November–December (Atia et al. 2011).

In many countries, sea fennel is traditionally used in cuisine as a fresh ingredient due to its interesting sensory attributes (Renna and Gonnella 2012), owing to its richness in essential oils (Özcan et al. 2001; Pateira et al. 1999). The fresh leaves can be used for preparing salads, soups and sauces, or they are kept like capers in vinegar; this latter food preparation is listed as a traditional agri-food product of Puglia (Southern Italy) by the Italian Ministry of Agriculture. In British Isles, "Rock Samphire Hash" is a traditional recipe that is prepared by mixing stems and leaves of *C. maritimum* L. with a pickled cucumber and caper. The food use of sea fennel is lost in the mists of time; according to a Greek legend, it is mentioned as a vegetable that was served to theseus by Hekate (Franke 1982). Sea fennel has been largely considered also for nutritional and medicinal purposes, since its leaves are rich in several compounds such as vitamin C, carotenoids, flavonoids as well as bioactive substances. Its seeds contain also appreciable amounts of edible oil, rich in essential fatty acids (Zarrouk et al. 2004). In former times sea fennel was used in folk medicine for its stimulating, diuretic and vermifuge effects. Sailors were used to eating fresh leaves as a protection against scurvy during fishing trips. In Italy, the sea fennel decoction was used against cystitis, prostatitis and colics, while the infusion was used in case of digestive diseases (Atia et al. 2011).

Sea fennel is a perennial halophyte typical of the Mediterranean, Pacific and Atlantic coasts (Atia et al. 2011; Perrino and Signorile 2009; Perrino et al. 2013); interestingly, its growth is stimulated by low salinity. Therefore, this wild edible plant may be considered as a promising crop in the context of biosaline agriculture (Atia et al. 2011). Unfortunately, sea fennel is currently an underutilized crop for commercial cultivation given its still relatively low global production and market value. This because this species may be cultivated globally, but is restricted to a more local production and consumption system. Probably, this can be ascribed also to the too low chance to obtain a real income due to the shortage of the consumer demand as fresh herb. Some Authors emphasize benefits from the food use from a wide availability of genetic resources such as wild edible plants (Hadjichambis et al. 2008; Pereira et al. 2011; Renna et al. 2015; Pignone and Hammer 2016). Therefore, the sea fennel, like other underutilized vegetables, by appropriate postharvest techniques could ensure consumer satisfaction and income within a more diversified and sustainable food production system (Ebert 2014).

Being aromatic herbs, sea fennel plants may be used not only as fresh product but also as dried herbs. In this context, food industry has developed a huge market for dried herbs which can be used either as single ingredients or for obtaining spice blends (i.e. curry powder) and seasoning blends. Blends also use visual attributes to grab attention, thus the introduction onto the market of a new dried herb, appreciable for both aroma and colour may be a way to improve the range of food. Although Renna and Gonnella (2012) reported the culinary use of dried sea fennel in some recipes, there is no current market for this commercial dried product. There is also a lack of information in the literature regarding technical processes for obtaining sea fennel powder, its potential acceptability by consumers as well as the evaluation of its colouring power.

It is well known that the quality of vegetables decreases after drying (Shin et al. 2015; Sahoo et al. 2015). On the other hand, while freeze-drying can minimize losses of flavour and aromatic compounds, it is one of he most expensive drying processes (Dalgleish 1990). Microwave-drying has been applied as an alternative method of dehydration because it is rapid and energetically efficient and provides high-quality dried foodstuffs (Chandrasekaran et al. 2013). Moreover, combined drying methods can be described as two or more drying methods combined in order to synthesize the advantages of each drying method and obtain high-quality products. This involves the use of one drying method, followed by one or more different drying methods (Huang et al. 2015).

On the basis of these knowledges, the aim of the present study was to apply different drying treatments on sea fennel for evaluating quality traits, consumer acceptability and colouring power of the dried powder. The general goal was to contribute for a better exploitation of this underutilized species by stimulating also commercial cultivation.

Materials and methods

Plant material and processing

Aerial parts of sea fennel were randomly collected from many plants along the shoreline near Mola di Bari (41°03′01″N 17°06′45″E) (Southern Italy). The harvested material was transferred to the laboratory and, after removing any inedible parts, was washed with tap water and blotted dry with paper towels. The edible material thus obtained was then mixed well, with 6300 g being divided up into seven portions (900 g per application). One portion was retained fresh, while the others were dried. Three treatment series per application (about 300 g of fresh vegetable) were prepared in order to provide independent replicates for each drying treatment and for the fresh samples. One-third of each replicate was used to extract essential oils, while the remaining portions of each replicate were used for sensory evaluation and for chemical and colour analysis. The used drying processing were:

- *Conventional air-drying* (AD) a forced-ventilation oven (Model M40-VF, MPM Instruments s.r.l., Bernareggio, Italy) was used to achieve three different processing temperatures. Therefore, fresh samples were air-dried at 45 °C (AD45), at 60 °C (AD60) and at 75 °C (AD75).
- *Microwave-drying* (MW) a programmable microwave oven (Kennex AG925CTW, Italy) was used, with maximum output of 900 W and internal volume of 25 L. One dish containing a single sample of fresh leaves was placed on the centre of a turntable inside the microwave cavity and processed for 2 min at 780 W, followed by 3 min at 450 W and by 10 min at 270 W.
- *Microwave-assisted air-drying* (MWAD) in this treatment, a combination of microwave- and air-drying (MWAD) was investigated in place of conventional air-drying to reduce dehydration time, according to Schiffmann (1992). Therefore, fresh samples were firstly dried by microwaving until a weight loss of 50% was achieved, and then by conventional air-drying at 45 °C.

- *Freeze-drying* (FD) a laboratory freeze-dryer (LABCONCO FreeZone® Freeze Dry System, model 7754030, Kansas City, USA) equipped with a stoppering tray dryer (LABCONCO FreeZone® Stoppering Tray Dryer, model 7948030, Kansas City, USA), was used.

Drying times were determined by preliminary tests, for each treatment, in order to obtain a final product with a water activity (a_w) value <0.6 (Darriet 2007; Muggeridg et al. 2000). Thus, based on the results obtained with the preliminary tests, for all treatments, the minimum drying time to obtain a weight loss of between 84 and 86% (Table 1) was applied. After each drying treatment, the dehydrated material was ground and passed through a fine-mesh sieve to obtain a powder with a diameter ≤1 mm. The powdered sea fennel was then packed in glass jars closed with an air-tight cap and stored at −20 °C without light, before being used for analysis and sensory evaluation. The total drying times for each treatment are presented in Table 1.

Measurement and analysis

For the calculation of weight loss, samples were weighed individually before and after each drying treatment. Results were expressed as % weight loss compared with initial raw material weight and utilized for expressing essential oils, chlorophyll and total carotenoid content on a fresh-weight basis. Water activity (a_w) of each dried sample was measured at 25 °C with a water activity meter (AquaLab Series 3, Decagon Devices Inc., USA). For the recovery of the essential oils fresh and dried samples were placed in a 0.25-L round-bottomed flask with 150 mL distilled water and separately steam-distilled for 4 h in a Clevenger-type apparatus, according to International Organization for Standardization (ISO) standards (1997). Chlorophylls content were determined spectrophotometrically using the extraction procedure reported by Bonasia et al. (2013) with some modifications: fresh and dried samples were homogenized in 80% acetone; the absorbance of the extract was measured at 647 and 664 nm, using a UV-1800 spectrophotometer (Shimadzu, Kyoto, Japan). For the colour measurements a colorimeter (CR-400, Konica Minolta, Osaka, Japan) equipped with illuminant D65, in reflectance mode and with the CIE L^* (lightness) a^* (redness) b^* (yellowness) colour scale, was used for the determination of the surface colour of fresh and dried samples. Before the readings were performed, the colorimeter was calibrated with a standard reference with L^*, a^* and b^* values of 97.55, 1.32 and 1.41, respectively. Hue angle ($h° = \text{arctg}\ b^*/a^*$) and saturation ($C = [a^{*2} + b^{*2}]^{1/2}$) were then calculated from primary L^*, a^* and b^* readings. Moreover, rice cream for adding the powdered sea fennel obtained with the different treatments was selected as a model system for evaluating the colouring power of the dried products. For each sample 1 g of powdered sea fennel was mixed with 100 g of rice cream. So, L^*, a^* and b^* readings were effected both on uncoloured rice cream and coloured samples in order to calculate the saturation (C) and Total Colour Difference (TCD). This latter parameter, used to indicate the magnitude of colour difference between

Table 1 Drying variables, weight loss (±SD) and water activity (±SD) of dried sea fennel for each treatment

Treatment	Drying variables		Weight loss (%)	Water activity
	Method	Time		
AD45	Air oven drying at 45 °C	72 h	85.16 ± 0.25	0.325 ± 0.007
AD60	Air oven drying at 60 °C	48 h	85.55 ± 0.15	0.166 ± 0.012
AD75	Air oven drying at 75 °C	24 h	85.49 ± 0.19	0.147 ± 0.014
MW	Microwaving	15 min	84.19 ± 0.23	0.516 ± 0.016
MWAD[a]	Microwaving air drying at 45 °C	7.5 min 24 h	84.12 ± 0.15	0.537 ± 0.012
FD	Freeze-drying	72 h	85.05 ± 0.32	0.326 ± 0.009

h hours, *min* minutes

[a] Samples were firstly treated by microwaving and then by air at 45 °C

uncoloured and coloured samples, was calculated according to Renna et al. (2014). For the sensory evaluation a selected group of 20 assessors (10 females and 10 males aged between 20 and 47 years), previously involved as members of a trained descriptive analysis panel for vegetables, was trained to evaluate the attributes of sea fennel. Colour, odour and overall acceptability of samples were evaluated using a hedonic scale ranging from 9 = highly acceptable to 1 = highly unacceptable (Renna et al. 2013). For data analysis the effect of different drying methods was tested by performing a one-way analysis of variance (ANOVA) using SAS software, with data means arranged in a completely randomized design. The means were separated by the Student–Newman–Keuls (SNK) test, while standard deviation (SD) was also calculated for data. In the experimental trials, three replications for each drying treatment were used.

Results and discussion

Water activity (a_w) of dried samples

Water activity and drying times for each treatment are reported in Table 1. Water activity is a useful parameter for determining the water availability of foods, constituting a critical approach for assessing food stability (Labuza 1997). The level of 0.6 a_w is generally accepted as the limit below which mould or microbial growth cannot occur (Muggeridg et al. 2000). Effectively, the minimum a_w for growth of most bacteria is approximately 0.87, although halophilic bacteria can grow at a_w as low as 0.75. While some species of xerophilic spoilage moulds and osmophilic yeasts can grow at a_w 0.60–0.70. The lowest a_w values were found in AD60 and AD74, while MW and MWAD samples showed the highest a_w levels. In each case, water activity was lower than 0.6, though it is important to underline that only MW resulted in dried sea fennel with good levels of a_w in a short time.

Essential oil content

Figure 2 shows the content of essential oils recovered from fresh and dried samples of sea fennel. In each case, the drying treatment significantly reduced the content compared to fresh samples. More specifically, the highest reduction (81%) was detected in AD75 samples, while the lowest (60% on average) in AD45, MWAD and FD samples. These results are in agreement with Rohloff et al. (2005), who reported a decrease in essential oil content in air-dried peppermint when the temperature increased from 30 to 70 °C. Similar results on air-dried basil were shown by Carvalho Filho et al. (2006), who found that samples dried at lower temperatures lost smaller quantities of essential oils. Moreover, Di Cesare et al. (2003) reported better retention of characteristic volatile compounds from essential oils in freeze-dried basil compared to microwaved and air-dried samples.

To our knowledge, these are the first data on the essential oil content from sea fennel affected by different drying treatments. In this study, freeze-drying and air-drying at a low temperature generally resulted in lower essential oil losses even when air-drying was microwave-assisted. Nevertheless, MW processing applied in this study made it possible to cut down drying times in comparison to all other drying treatments, without significantly reducing the content of essential oils.

Chlorophyll content

The effects of drying processing on chlorophylls is reported in Table 2. As in the case of essential oils, all drying treatments significantly reduced chlorophyll content. On average, AD60 and AD75 air-drying methods resulted in the highest reduction (67, 79 and 70%, for chlorophyll *a*, chlorophyll *b* and total chlorophylls, respectively). AD45, compared to AD60 and AD75, caused lower reductions in chlorophyll *b* and total chlorophylls, while it had similar effects on chlorophyll *a* content (Table 2). MWAD reduced chlorophyll *b* content to the same extent as AD45, but influenced total and chlorophyll *a* less than conventional air-drying methods. At the same time, the lowest reduction for all pigments was detected in MWAD as well as in MW and FD (on average, 33, 47 and 42% for chlorophyll *a*, chlorophyll *b* and total chlorophylls, respectively). Chlorophylls are abundant pigments in green vegetables, strongly related to the colour characteristics and their content is an important quality parameter, reflecting the appearance of the final dried product (Shin et al. 2015). This study's results relating to chlorophylls are in agreement with Kathirvel et al. (2006), who reported a lower

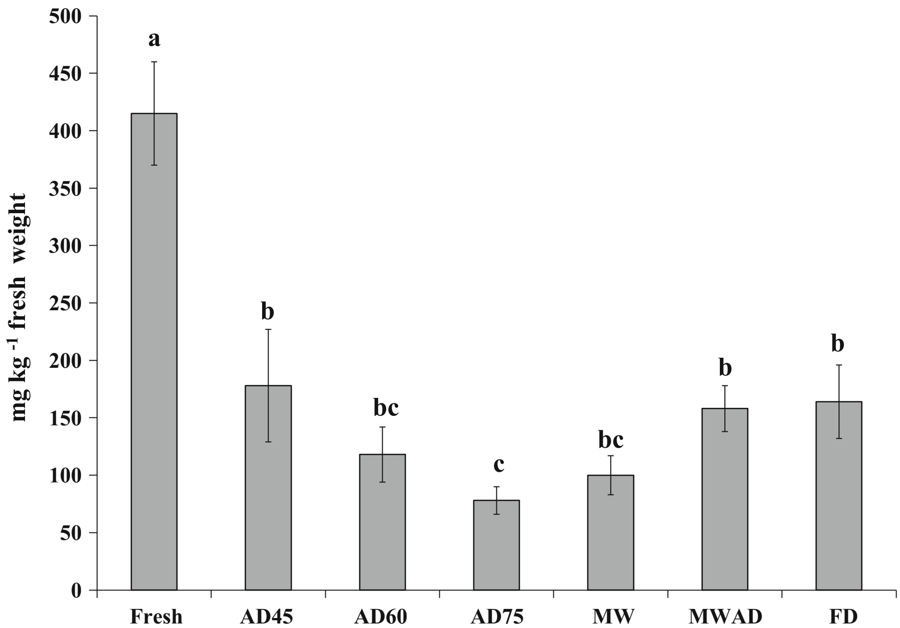

Fig. 2 Essential oils content in fresh and dried samples of sea fennel. *AD45* air oven drying at 45 °C, *AD60* air oven drying at 60 °C, *AD75* air oven drying at 75 °C, *MW* microwave drying, *MWAD* microwave-assisted air oven drying, *FD* freeze-drying. Significance: $P \leq 0.001$. The *same letters* indicate that mean values (n = 3) are not significantly different ($P = 0.05$)

Table 2 Chlorophyll content (±SD) in fresh and dried samples of sea fennel

	Chlorophyll *a* (mg 100 g^{-1} fresh weight)	Chlorophyll *b* (mg 100 g^{-1} fresh weight)	Total chlorophylls (mg 100 g^{-1} fresh weight)
Fresh	55.49 ± 8.09 a	18.17 ± 3.01 a	73.66 ± 11.10 a
AD45	23.20 ± 0.19 c	7.69 ± 0.06 c	30.89 ± 0.15 c
AD60	18.89 ± 0.83 c	3.50 ± 0.20 d	22.38 ± 1.00 d
AD75	18.23 ± 0.42 c	3.97 ± 0.15 d	22.20 ± 0.57 d
MW	34.02 ± 0.66 b	9.99 ± 0.33 b	44.01 ± 0.92 b
MWAD	31.98 ± 0.55 b	9.15 ± 0.13 bc	41.13 ± 0.67 b
FD	32.71 ± 3.59 b	9.75 ± 0.86 bc	42.46 ± 4.44 b
Significance	***	***	***

AD45 air oven drying at 45 °C, *AD60* air oven drying at 60 °C, *AD75* air oven drying at 75 °C, *MW* microwave drying, *MWAD* microwaving + air oven drying, *FD* freeze-drying

*** Significant for $P \leq 0.001$. The same letters in the same column indicate that mean values (n = 3) are not significantly different ($P = 0.05$)

chlorophyll decrease in coriander, mint and parsley dried by microwaving in comparison with air-dried samples. Moreover, they found that chlorophyll reduction was generally higher in samples dried at 75 °C compared to those dried at 45 °C. So, the chlorophyll decrease observed in air-dried sea fennel was probably due to thermal damage, which increased with drying temperature.

Surface colour of sea fennel

Table 3 shows the surface colour parameters for fresh and dried sea fennel, while the visual appearance of all these samples is shown in Fig. 3. FD caused the highest increase in *L**, while AD60, AD75 and MW did not show significant differences compared to fresh samples. Moreover, AD45 caused a significant

Table 3 Surface CIE *L*a*b** color parameter in fresh and dried samples of sea fennel

	*L**	*a**	*b**	h°	C
Fresh	41.51 ± 0.83 d	−11.66 ± 0.45 b	16.27 ± 0.56 f	125.62 ± 0.13 a	20.01 ± 0.71 f
AD45	45.35 ± 0.27 b	−7.78 ± 0.28 b	31.07 ± 0.07 c	104.06 ± 0.46 d	32.03 ± 0.13 c
AD60	40.85 ± 0.25 d	−0.59 ± 0.05 f	23.38 ± 0.20 e	91.45 ± 0.14 f	23.39 ± 0.20 e
AD75	41.48 ± 0.33 d	−3.76 ± 0.12 e	26.14 ± 0.24 d	98.18 ± 0.27 e	26.40 ± 0.23 d
MW	41.03 ± 0.16 d	−15.87 ± 0.14 a	34.57 ± 0.32 a	114.66 ± 0.16 b	38.04 ± 0.34 a
MWAD	43.52 ± 0.49 c	−10.93 ± 0.18 c	32.20 ± 0.28 b	108.75 ± 0.39 c	34.01 ± 0.23 b
FD	52.35 ± 0.64 a	−15.94 ± 0.22 a	34.39 ± 0.17 a	114.87 ± 0.20 b	37.90 ± 0.24 a
Significance	***	***	***	***	***

*L** lightness, *a** redness, *b** yellowness, *h°* hue angle, *C* saturation

AD45 air oven drying at 45 °C, *AD60* air oven drying at 60 °C, *AD75* air oven drying at 75 °C, *MW* microwave drying, *MWAD* microwave-assisted air oven drying, *FD* freeze-drying

*** Significant for $P \leq 0.001$. The same letters in the same column indicate that mean values (n = 3) are not significantly different ($P = 0.05$)

increase in *L**, which was lower than the value for FD but higher compared to MWAD. These results are in agreement with Di Cesare et al. (2003), who reported the highest *L** value for freeze-dried basil, while similar *L** values were observed between microwaved samples and fresh basil. Moreover, Yousif et al. (2000) showed the highest *L** value in freeze-dried Mexican oregano (*Lippia berlandieri* Schauer) in comparison with other drying methods.

As regards h°, all treatments caused significant reductions. However, in MW and FD samples h° decreased by 8.6%, while the reduction was 13.4, 17.2, 21.8 and 27.2%, respectively in MWAD, AD45, AD75 and AD60. These results suggest that FD and MW reduce the colour changes in the dried samples compared to other drying methods, generating a "green colour" which may be perceived as more similar to that of fresh sea fennel. By contrast, higher temperatures during air-drying can cause greater colour changes in dried samples compared with fresh sea fennel. According to some authors (Soysal 2004; Oliveira et al. 2015), these results indicate that increasing drying temperatures reduces the intensity of the "green colour" by reducing chlorophylls to a greater degree (Table 2).

Finally, as regards the C value (which indicates the degree of difference of a hue in comparison to a grey colour with the same lightness) all the treatments caused a significant increase, which was higher in FD and MW samples. Indeed, an increase of about 89.7% was found in these samples, while in MWAD, AD45, AD75 and AD60 the increase was 70.0, 60.1, 30.1 and 15.4%, respectively. The higher the C values, the higher the colour intensity of samples perceived by humans (Pathare et al. 2013). Therefore, this quantitative attribute of colourfulness may have influenced the panellists during sensory evaluation, as reported below.

Colouring power evaluation

Figure 4 reports the saturation (C value) and Total Colour Difference (TCD) as colouring power indicators of powdered sea fennel samples in rice cream. As regards saturation, the rice cream coloured by the FD sample showed the most vivid colour, consisting of a C value over sixfold higher than uncoloured rice cream. Differently, the rice cream coloured with MW, MWAD and AD45 samples, showed a saturation of about 5.4-fold, 4.3-fold and 3.7-fold higher than uncoloured rice cream, respectively. The samples coloured by AD60 and AD75 sea fennel showed the lowest saturation. Similarly to saturation, the highest value of TCD was found in FD rice cream, followed by the sample coloured by MW sea fennel. In all the other cases, TCD was lower, especially for the sample coloured by AD60. These results show that in order to obtain the highest increase in saturation and TCD, FD powdered sea fennel should be used or, as an alternative, the dried product obtained by microwaving.

Natural food colorants have become increasingly popular with consumers because the synthetic

Fig. 3 Fresh and dried samples of sea fennel

alternatives tend to be perceived as undesirable and harmful. Moreover, they have received a particular attention also for conferring functional properties to food products. To this regard, natural food colorants could make it possible to ensure the consumer satisfaction since they are considered not only as organoleptic improvement ingredients, but also health promoters and enhancers of nutritional status (Martins et al. 2016). Many authors report the use of vegetables and their extracts as natural sources of food colorants (Martínez et al. 2006; Duangmal et al. 2008; Díaz-García et al. 2015; Martins et al. 2016). In our previous study (Renna and Gonnella 2012), freeze-dried sea fennel was used to obtain green *tagliatelle* (a type of Italian pasta). Thus, the results of the present study underline the possibility to use dried sea fennel for its colouring properties in another food model, preferably as a freeze-dried product. Therefore, we could consider powdered sea fennel as a product usable not only for its aromatic traits due to the richness in essential oil but also for its colouring power as for other natural food colorants from plant origin such as *Crocus sativus* L., *Gardenia jasminoides* Ellis, *Genipa americana* L., *Rubia tinctorum* L., etc. (Martins et al. 2016).

Sensory evaluation

The sensory profile of fresh and dried sea fennel is shown in Fig. 5. The dried samples differed significantly in colour, odour and overall acceptability. As regards colour, the panellists preferred the MW

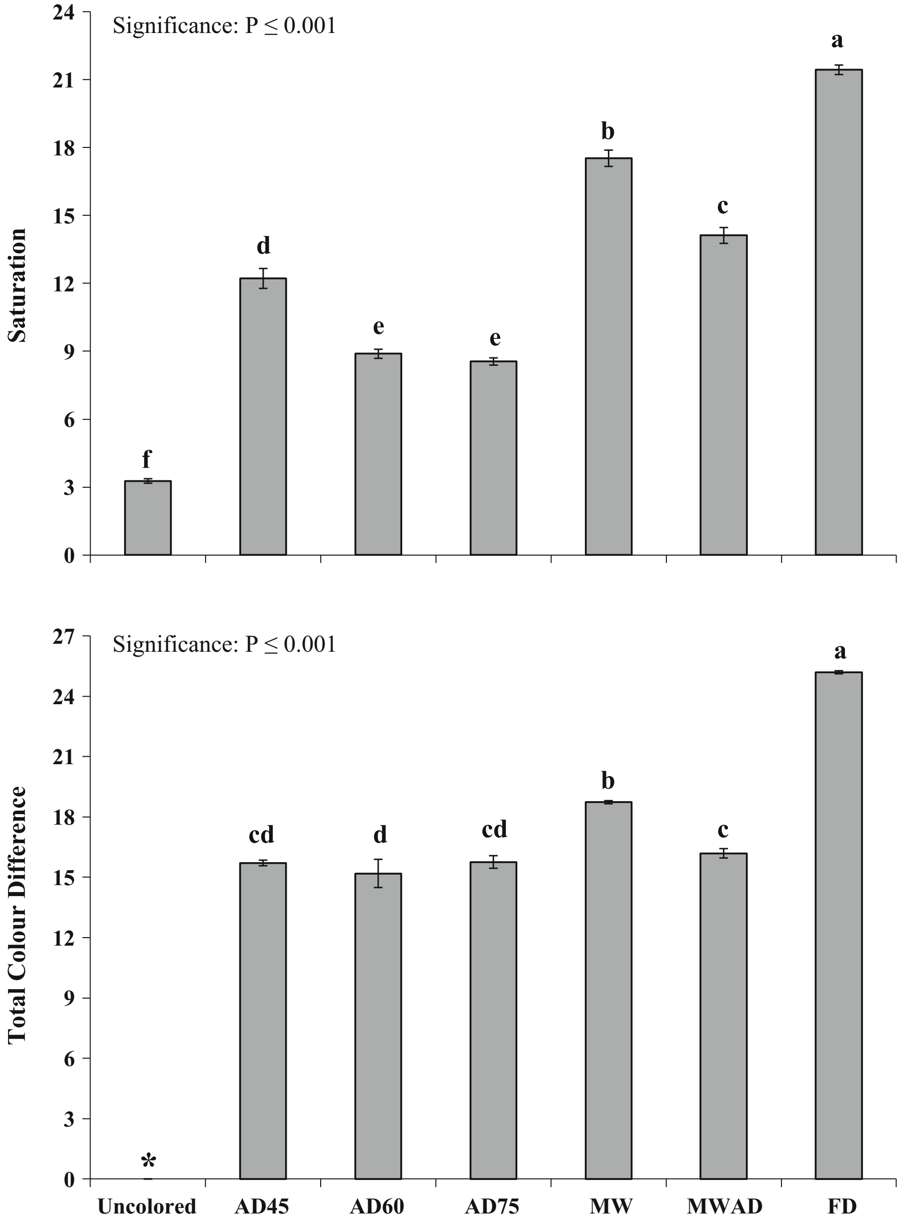

Fig. 4 Saturation and total *color* difference of the *uncolored* rice cream and samples *colored* by sea fennel powder. *AD45* air oven drying at 45 °C, *AD60* air oven drying at 60 °C, *AD75* air oven drying at 75 °C, *MW* microwave drying, *MWAD* microwave-assisted air oven drying, *FD* freeze-drying. TCD values were calculated as the difference between the *color* parameters in the *uncolored* rice cream (*TCD = 0) and samples *colored* by powdered sea fennel. The *same letters* indicate that mean values (n = 3) are not significantly different ($P = 0.05$)

sample (8.1), whereas AD60 and AD75 were not acceptable (3.7 on average). At the same time, AD45 was slightly acceptable (5.5), while for FD and MWAD the panellists placed them in the "like moderately" category (6.8 on average) (Fig. 5). In agreement with the h° values obtained by colorimeter, these results show that the panellists appreciated significant differences between MW sea fennel and other dried samples. However, the lower colour score attributed to the FD sample by panellists is probably due to its greater L^* value (Table 3). So, it is possible that a high L^* value could reduce visual perception of

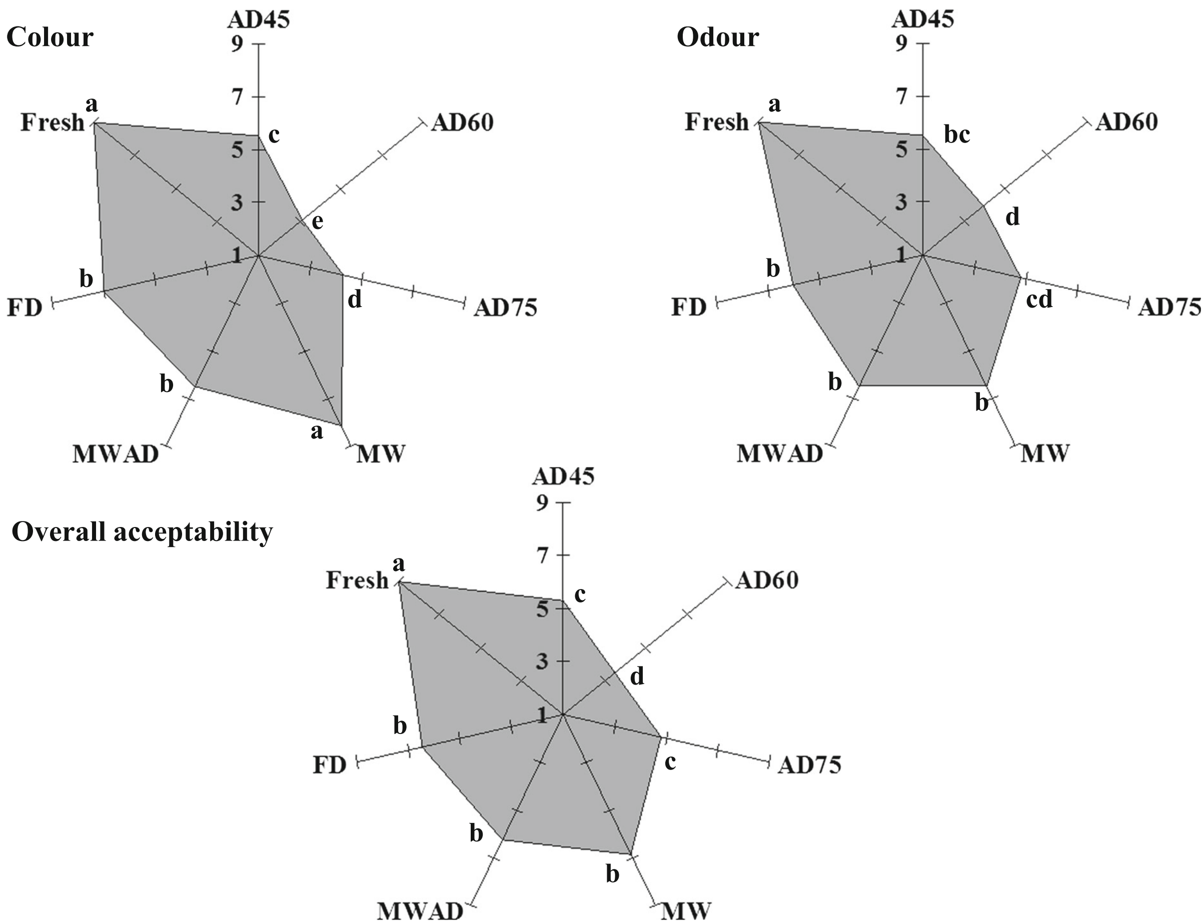

Fig. 5 Sensory evaluation (scale 1–9) for fresh and dried samples of sea fennel. *AD45* air oven drying at 45 °C, *AD60* air oven drying at 60 °C, *AD75* air oven drying at 75 °C, *MW* microwave drying, *MWAD* microwave-assisted air oven drying, *FD* freeze-drying. Significance: $P \leq 0.001$. The *same letters* in the same column indicate that mean values (n = 60) are not significantly different ($P = 0.05$)

the colour in the case of similar h° values. At the same time, the lowest score attributed to AD60 and AD75 samples is in agreement with their considerable reduction in h° values compared to fresh sea fennel (Tab. 4). Therefore, it is possible that the greater change in h° displayed by these dried samples compared to fresh sea fennel was not well appreciated by panellists.

As regards odour in all dried samples, the score was significantly lower compared to fresh sea fennel. Nevertheless, the panellists preferred FD, MWAD and MW (6.3 on average) compared to unacceptable AD60 and AD75 (4.3 on average) (Fig. 5). Moreover, AD45 (5.5) was significantly different from fresh sea fennel and AD60, but not significantly different from the other dried samples. These results are in agreement with the essential oil content reported above (Fig. 2). Indeed, in all dried samples, the content of essential oils was significantly lower compared to fresh sea fennel. Therefore, the lower odour score of all dried samples is probably due to their significantly lower content in essential oils (Fig. 2). Nevertheless, the lowest odour score of AD60 is not attributable only to the essential oil content, since this was not significantly different compared to the other dried samples (Fig. 2). Rather, it is likely that odour perception is affected by several factors such as the possible production of off-flavours during conventional air-drying.

Finally, FD, MWAD and MW got the highest overall acceptability rating for dried sea fennel (6.5 on average), while AD60 got the lowest (3.5). At the same time, the panellists neither liked nor disliked AD45 and AD75 (5.0 on average) (Fig. 5). On the whole, as regards powdered sea fennel, the panellists preferred FD and microwave-treated samples, while all the samples dried using only hot air got the lowest average score. Therefore, in order to obtain a product that is also appealing to consumers, it is important to consider the effects of drying on sensory attributes, since they could represent important drivers of acceptance for consumers.

Conclusions

Many underutilized crops are already cultivated but are undervalued given their still relatively low global production and market value. Some of these underutilized species may be widely distributed globally but are restricted to a more local production and consumption system. In this study, a new food product was obtained by drying sea fennel using different treatments for promoting the consumption of this underutilized species and to stimulate commercial cultivation. Experimental results from the quality assessment and consumer acceptability indicate that it is possible to consider dried sea fennel as a product usable not only for its aromatic traits but also for its colouring power as for other natural food colorants from plant origin. Drying processes of the present work may be used for the industrial production on a large scale and also to diversify local food through a micro-scale production. It could be concluded that, this underutilized crop could play a better role for making up a sustainable food production system. Nevertheless, more agronomic information could be needed before any large-scale cultivation of sea fennel for obtaining a sustainable better exploitation of this promising species. Finally, possible next goals may be directed toward the changes assessment of the nutritional and functional sea fennel components during the dehydration processes.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest in connexion to the present manuscript.

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Hot air and freeze-drying of high-value foods: a review

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Abstract

Drying is an ancient process used to preserve foods. Conventional drying (hot air) offers dehydrated products that can have an extended life of a year. Unfortunately, the quality of a conventionally dried product is drastically reduced from that of the original foodstuff. Freeze-drying is based on the dehydration by sublimation of a frozen product. Due to the absence of liquid water and the low temperatures required for the process, most of deterioration and microbiological reactions are stopped which gives a final product of excellent quality.

The comparison of both preservation processes, hot air and freeze-drying, was done taking into account several important characteristics such as shrinkage, glass transition temperature, process–quality interaction, drying kinetics, costs and new improvements. An updated bibliographic research served to compare both drying processes. Experimental data as well as theoretical results, from several years of research in the subject, were presented and compiled in order to support conclusions. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: High-value foods; Freeze-drying; Hot air-drying; Quality

1. Introduction

According to the Longman Dictionary of Contemporary English (1990), a food can be defined as something that living creatures take into their bodies to provide them with energy and to help them to develop and to live. Among the great variety of foods currently available, a high-value food can be defined as the one that naturally has above the average worth as compared to others. Many examples representing high-value foods can be mentioned: (a) seasonal and perishable commodities, due to their limited availability; (b) baby foods, since is desirable to feed them with maximum quality and nutritional foods; (c) nutraceutical foods; (d) distinguished organoleptical foods, such as aromatic herbs or coffee; and (e) special end use foods, as those used for outdoor activities, military rations or instant meals.

It is well known that processes may affect (partially or totally) the quality of a product. Indeed, various changes in physical, chemical and/or biological characteristics of foodstuffs may occur during processing, storage and distribution (Karel, Buera, & Roos, 1993). These changes alter the physical aspect such as color and structure. They can also develop undesirable biochemical reactions such as deterioration of aroma compounds or degradation of nutritional substances (Achanta & Okos, 1995; Chirife & Buera, 1995; Karel, 1991; Karmas, Buera, & Karel, 1992; Roos & Himberg, 1994; Roos & Karel, 1991; Sapru & Labuza, 1993; Stapelfeldt, Nielsen, & Skibsted, 1997). All the fore-mentioned physical and biochemical changes certainly cause a reduction in product quality and in process efficiency as well (Chuy & Labuza, 1994). Particularly when dealing with high-value foods, the choice of the right method of preservation can therefore, be the key for a successful operation.

The term drying refers generally to the removal of moisture from a substance. It is the most common and most energy-consuming food preservation process. With literally hundreds of variants actually used in drying of particulate solids, pastes, continuous sheets, slurries or solutions, it provides the most diversity among food engineering units operations (Ratti & Mujumdar, 1995). Air-drying, in particular, is an ancient process used to preserve foods in which the solid to be dried is exposed to a continuously flowing hot stream of air where moisture evaporates. The phenomena underlying this process is a complex problem involving simultaneous mass and energy transport in a hygroscopic, shrinking system. Air-drying offers dehydrated products that can have an extended life of a year but, unfortunately, the

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Nomenclature

a_w	water activity, dimensionless
Bi_{md}	Biot mass number in the dried zone, dimensionless
C	color saturation degree
C_1	universal constant for the WLF equation
C_2	universal constant for the WLF equation
D_r	water diffusivity
k	kinetic constant for NEB browning
k_g	mass transfer coefficient
L_o	characteristic length
n_w	water flux
P_1	equilibrium relationship at the interface
p_w	water vapor pressure
p_{ws}	water vapor at saturation
T	temperature
T_g	glass transition temperature
X	water content, dry basis
Φ	characteristic drying-curve parameter (Ratti & Crapiste, 1992)
Subscripts	
o	initial
ref	at reference temperature bulk air

quality of a conventionally dried product is usually drastically reduced from that of the original foodstuff.

Vacuum freeze-drying is the best method of water removal with final products of highest quality compared to other methods of food drying (Genin & René, 1995; Irzyniec, Klimczak, & Michalowski, 1995). Freeze-drying is based on the dehydration by sublimation of a frozen product. Due to the absence of liquid water and the low temperatures required for the process, most of deterioration and microbiological reactions are stopped which gives a final product of excellent quality. The solid state of water during freeze-drying protects the primary structure and the shape of the products with minimal reduction of volume. Despite of many advantages, freeze-drying has always been recognized as the most expensive process for manufacturing a dehydrated product.

The aim of this paper is to compare both preservation processes, hot air and freeze drying, in terms of process-quality interaction, drying kinetics, costs and new improvements, in order to see their applicability to high-value foods.

2. Materials and methods

A bibliographic research was done using the following data bases: Agro-Base (1970–1998), FSTA (1970–1998) and Current Contents. Experimental data as well as theoretical results, from several years of research in the subject, are presented and compiled in order to support conclusions.

3. Discussion

3.1. Quality and glass transition

Since the last decade, quality of foods became among the main preoccupations in food research. The application of the knowledge learned in the area of glass transition of polymers to food systems had success in understanding and predicting the behavior of foodstuffs (Schenz, 1995). Glass transition temperature, T_g, can be defined as the temperature at which an amorphous system changes from the glassy to the rubbery state (Karmas et al., 1992; Roos & Karel, 1991b). This parameter can be determined experimentally by following the variation of some physic, thermodynamic or dielectric properties as a function of temperature (Roos, 1992; Slade & Levine, 1991; Roos, 1995). Recently, glass transition temperature of food products has been pointed out to be responsible for the deterioration mechanisms during processing, and an indicator of food stability (Chirife & Buera, 1995; Roos, 1995; Slade & Levine, 1991; Kerr, Lim, Reid, & Chen, 1993; Roos & Karel, 1991; Schenz, 1995; Peleg, 1995). It has been also reported that when temperature of some processes exceeds T_g, the quality of foodstuffs is seriously altered (Peleg, 1996). Unfortunately, quantifiable expressions between quality parameters and glass transition have not been found yet. Process-quality relationships could be a solid basis to optimize both existing and novel dehydration methods (Nijhuis et al., 1996).

Research advances on the effect of drying and freeze-drying on three quality parameters (rehydration, color and volume) as well as the recent steps towards the understanding of the quality-glass transition relationship will be analyzed in this section. Fig. 1 presents the progression of number of scientific articles on the selected quality parameters as a function of the year of publication. As can be seen, while the attention on research about color deterioration and shrinkage during drying has been particularly focused in the last years, rehydration is the quality parameter the most studied in the literature. Indeed, no dried product can have a good quality if its rehydratability is low. Although numerous, most of the published articles just analyze the effect of drying pretreatment or drying conditions on rehydration (Rastogi & Niranjan, 1998; Hammami & René, 1997; Stute & Knorr, 1994; Garcia-Reverter, Bourne, & Mulet, 1994; etc.). More recently, the attention have been focused on the effect of drying methods (or combination of different methods) on rehydration (Pappas, Tsami, & Marinos-Kouris, 1999; Litvin, Mannheim, & Miltz, 1998; Lin & Durance, 1998; etc.). Rehydration ratio of

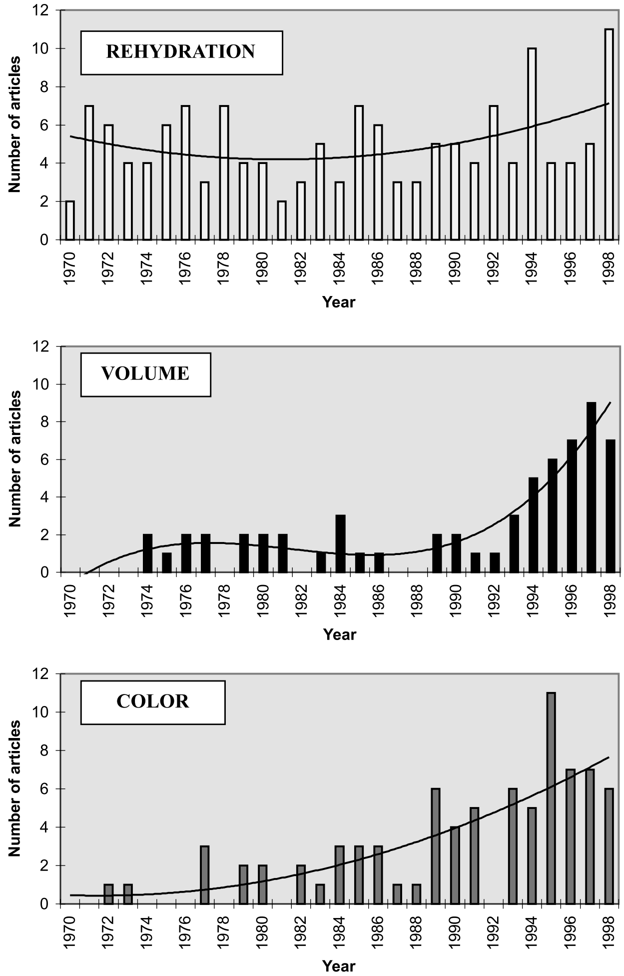

Fig. 1. Progression of the number of articles on quality parameters related to drying methods in the last 30 years.

freeze-dried foods is in general 4–6 times higher than air-dried foods, making freeze-dried products excellent for ready-to-eat instant meals or soups. The relationship between rehydration and glass transition temperature should be interpreted from porosity and collapse during drying or freeze-drying, which will be discussed later.

The studies on the deterioration of color during drying and freeze-drying of high-value foods have had an important significance not only in the assessing of their visual aspect but also because of the close relationship between antioxidant or vitamin contents and color. Fig. 2 shows a comparison of change in color (degree of saturation C) of pulp and skin of strawberries during air-drying and freeze-drying (Shishegarha & Ratti, 1999) at different operating temperatures. Discoloration and browning during air-drying are the result of various reactions including the pigment destruction (Garcia-Viguera et al., 1998; Krokida, Tsami, & Maroulis, 1998). High temperatures during drying were found to exert a great influence to degrade the color (Bakker, Bridle, & Koopman, 1992). On the other hand,

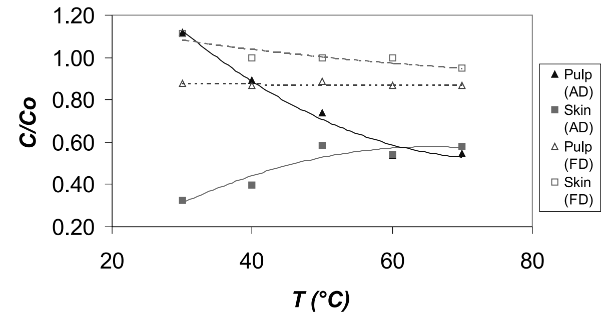

Fig. 2. Changes in color during air-drying and freeze-drying of strawberries at different temperatures (T represents air temperature for air-drying or heating plate temperature for freeze-drying, AD = air-drying and FD = freeze-drying).

the red color of strawberries was just slightly increased during freeze-drying. According to the statistical analyses there is no significant effect of the excessive heating up to 70°C on the changes of color parameters during vacuum freeze-drying (Shishegarha & Ratti, 1999). This can be explained with the low temperature inside product due to the poor internal heat transfer in the dry layer of product during freeze-drying process. The minimal color deterioration during freeze-drying is an indication of the appropriateness of this process to preserve nutraceutical foods.

Some works on the relationship of non-enzymatic browning (NEB) and glass transition have been presented in the literature (Roos & Himberg, 1994; Karel et al., 1993; Karmas et al., 1992; Flink, 1983). In order to evaluate the effect of temperature and glass transition on the rate of NEB near the zone of glass transition, the Williams–Landel–Ferry (WLF) equation (Williams, Landel, & Ferry, 1955) has been used to represent the kinetic NEB constant k.

$$\ln \frac{k_{\text{ref}}}{k} = \frac{-C_1(T - T_g)}{C_2 + (T - T_g)}. \tag{1}$$

It was concluded that WLF equation was not applicable to describe NEB if the experimental T_g value and the universal constants, C_1 and C_2 are used (Karmas et al., 1992). Nevertheless, if a reference temperature and adjustable parameters are used, this equation could be applicable to quantify the relationship between NEB and glass transition (Karel et al., 1993).

Considerable changes in the physical structure of product, such as reduction in volume and decrease in porosity can be found during drying (Jankovié, 1993; Ratti, 1994; Krokida & Maroulis, 1997). In freeze and spray-drying, for example, collapse is a frequent problem if certain operation variables are not well set (Roos, 1995; Slade & Levine, 1991). This phenomenon occurs when the solid matrix of the foodstuff can no longer support its own weight, leading to drastic structural changes shown as a marked decrease in volume, increase in stickiness of dry powders, loss of porosity, etc. (Chuy & Labuza, 1994; Levi & Karel, 1995; Roos, 1995). A comparison of the final volume reduction during both air and freeze-drying of berries is shown in Table 1 (Jankovié, 1993). As can be seen, shrinkage during freeze-drying is minimal (from 5% to 15%) while during air-drying is excessive (around 80%). This behavior has been observed for most types of foodstuffs (Ratti, 1994; Karathanos, Anglea, & Karel, 1993; Lozano, Rotstein, & Urbicain, 1983). In the case of air-drying, reduction of volume is usually accompanied by wrinkles, deformation, and even change in color, indicating the collapse of air-dried products. Freeze-drying under sub-optimal conditions can also lead to collapse (Levi & Karel, 1995). Indeed, the percentage of collapsed samples during freeze-drying can increase with heating plate temperature as shown in Fig. 3 for strawberries (Shishegarha & Ratti, 1999). Shrinkage and T_g are interrelated in that significant change in volume can be noticed only if the temperature of the process is higher than the T_g of the material at that particular moisture content. In effect, above T_g the viscosity drops considerably to a level that facilitates deformation (Genin & René, 1995; Levi & Karel, 1995; Roos & Himberg, 1994). In order to understand better the collapse phenomena, the progression of the product temperature during air and freeze-drying should be compared to its glass transition temperature at comparable moisture contents, as shown in Fig. 4 for the case of strawberries (air-dried at 50°C and freeze-dried at 20°C). In the case of air-drying, the product temperature is above T_g during the whole process, which demonstrates that collapse and poor quality should be expected when air-drying. On the other hand, and on the contrary to what could be initially expected, product freeze-drying temperatures are below T_g only at the end of the process. Nevertheless, to compare temperatures at the average moisture content of the product during freeze-drying would lead to a misunderstanding of the problem since, in this particular process, the solid is divided into dry and frozen regions separated by a receding front. The portion of the solid that is in contact with the highest temperatures for long periods of time is thus, the amorphous dry matrix, which has low moisture content and a T_g corresponding closely to that of the dry solids. The previous explanation and the fact that collapse is a time-temperature phenomenon (Gerschenson, Batholomai, & Chirife, 1981; Tsourouflis, Flink, & Karel, 1976) suggest that the glass transition temperature of dry solids (T_{gs}) would be a crucial optimization parameter for the freeze-drying process. This parameter may also serve as a useful tool for the choice of the most appropriate materials to be freeze-dried.

Table 1
Shrinkage of berries during air-drying and freeze-drying (from Jankovié, 1993)

	Volume reduction (%)	
	Freeze-drying	Air-drying
Strawberry	6.59	80.26
Raspberry	6.13	74.97
Blackberry	16.13	78.05

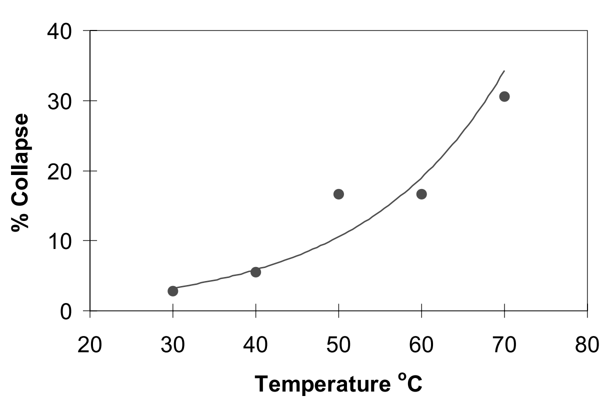

Fig. 3. Collapse percentage during freeze-drying of strawberries (Shishegarha & Ratti, 1999).

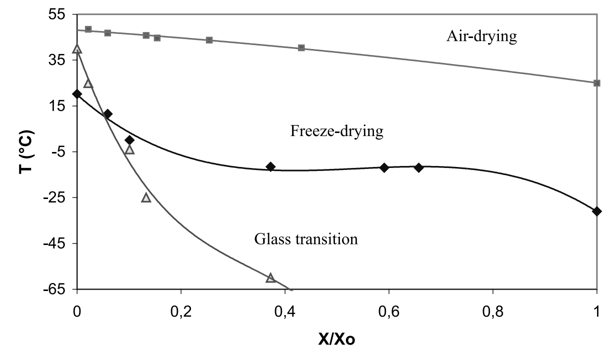

Fig. 4. Progression of temperatures during drying (at 50°C) and freeze-drying (at 20°C) of strawberries as compared to its glass transition temperature.

3.2. Drying kinetics

Drying kinetic of the product is the most important data required for the design and simulation of dryers. This essential information can be obtained through simple lab-scale experiments. Nevertheless, due to the uncertainty of the process, particularly when dealing with biological materials, all the necessary data under different operating conditions is not usually available.

Fick's law type models are commonly used to represent the air drying kinetics in the falling rate period. However, this simple model is not always adequate to represent the complex process of drying. Any simple theory idealizes the process and generally cannot be used safely for the design of dryers (Keey, 1980). On the other hand, there are some complex theories that represent the drying process from the microscopic standpoint of mass and heat transfer between each phase inside the food particle (Crapiste, Whitaker, & Rotstein, 1988). This careful approach, which uses the volume averaging method to solve the governing transport equations, is sometimes too complex for practical use since the required parameters are difficult or impossible to determine experimentally. For practical purposes, it is often useful to use a lumped-parameter model supported by carefully designed experimentation at laboratory scale.

Ratti and Crapiste (1992) developed a lumped parameter model for hygroscopic shrinking food systems. This model is represented by the following equation.

$$n_{\mathrm{w}} = \frac{k_{\mathrm{g}}(a_{\mathrm{w}}p_{\mathrm{ws}} - p_{\mathrm{w}\infty})}{[1 + (\Phi/X_{\mathrm{o}})Bi_{\mathrm{md}}]}, \qquad (2)$$

where Bi_{md} is the Biot number for mass transfer defined as $Bi_{\mathrm{md}} = (k_{\mathrm{g}}L_{\mathrm{o}})/(P_{\mathrm{l}}D_{\mathrm{r}}\rho_{\mathrm{s}})$ (Ratti & Crapiste, 1992; Ratti, 1994) and P_{l} is the equilibrium relationship at the solid–gas interface, which can be obtained from $P_{\mathrm{l}} = (\partial X/\partial p_{\mathrm{w}})_{\mathrm{T}}$.

The parameter Φ in Eq. (2) was shown theoretically and experimentally to be independent of drying conditions and particle geometry, and only a function of moisture content (Ratti & Crapiste, 1992). Fig. 5 shows the experimental value for Φ for different food products. The fitting of experimental data to a power function gives the following equation for several fruits and vegetables, and for various particle shapes (Ratti & Crapiste, 1992).

$$\Phi = 5.32 \times 10^{-3}(X/X_{\mathrm{o}})^{-1.079}. \qquad (3)$$

A short-cut method, based on the previous lumped parameter model, was developed to simulate air dying kinetic curves from a minimum number of experimental data (Fontaine & Ratti, 1999). A comparison between experimental and predicted (hot air) drying curves for

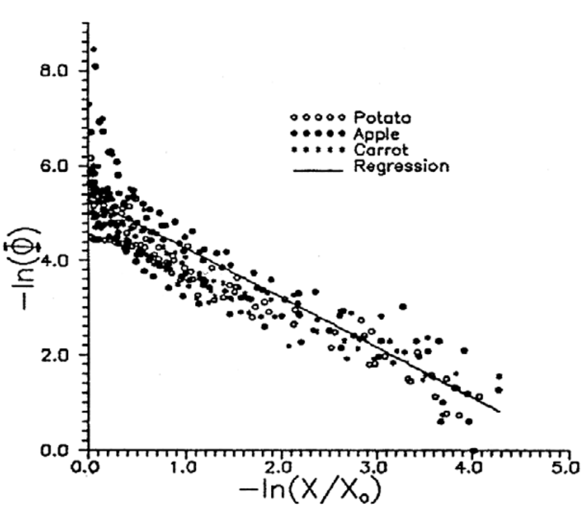

Fig. 5. Experimental parameter Φ (Ratti & Crapiste, 1992).

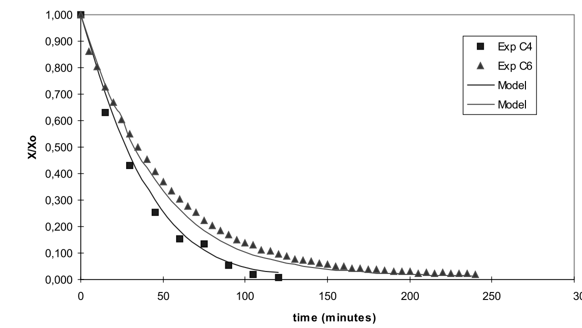

Fig. 6. Predictions of mushroom drying curves from a short-cut method as compared to experimental data (Fontaine & Ratti, 1999).

bananas, grapes and mushrooms revealed a close agreement for different operating conditions. Fig. 6 shows an example of the application of the short-cut method to the prediction of drying curves for mushrooms.

Recently, simulation has been used as preliminary evaluation of freeze-drying. Several theoretical models concerning the heat and mass transfer phenomena during the freeze-drying can be found in the literature (Lombraña, De Elvira, & Villarán, 1997; Lombraña & Izkara, 1996; Liapis & Bruttini, 1995a; Mellor, 1978; Karel, 1975). However, in most cases adjustable parameters are needed to match the model predictions with experimental data (Sheehan & Liapis, 1998; Sadikoglu & Liapis, 1997; Sharma & Arora, 1993; Millman, Liapis, & Marchello, 1985). In other cases, no comparison with experimental data is presented (Liapis & Bruttini, 1995b; Nastaj, 1991).

A new model has been built using mass and energy balances in the dried and frozen regions of the material undergoing freeze-drying (Khalloufi, Robert, & Ratti, 1999). Both sublimation and desorption are taken into account in the set of coupled non-linear partial differential equations. These equations were solved numerically by using a finite element scheme. The non-linear problem and the interface position were solved by the Newton–Raphson approach. All the parameters involved in the model (i.e., thermal conductivity, permeability, heat and mass transfer coefficients etc.) were obtained independently from experimental data cited in the literature. Simulation results agreed closely to experimental data as shown in Fig. 7 for apple freeze-drying. This model can be considered as a useful tool for optimization the freeze-drying process.

3.3. Costs

Freeze-drying is generally seen as a very expensive preservation method. Its cost varies depending on the type of raw material, the products, the packaging, the capacity of the plant, duration of cycle, etc. (Lorentzen, 1979; Sunderland, 1982a). As compared to air-drying,

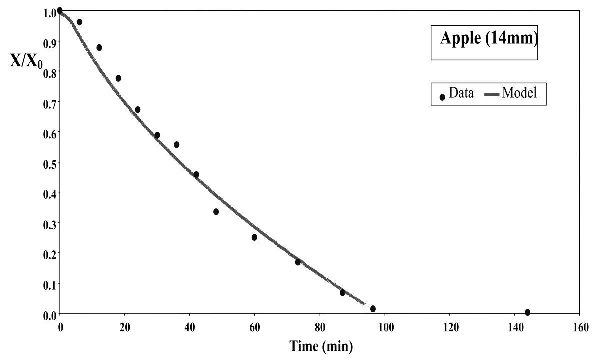

Fig. 7. Freeze-drying of apple, comparison between model predictions and experimental data (Khalloufi et al., 1999).

freeze-drying costs are 4–8 times higher (Flink, 1977a; Mafart, 1991). However, it is important to include all energy uses when evaluating or comparing different processes. For example, the comparison between freeze-drying cost to other methods of food preservation as freezing is quite advantageous if the energy spent at the home storage freezer is taken into account (see Table 2, Flink, 1977b; Judge et al., 1981).

Fig. 8 shows a comparison of freeze-drying costs associated to the processing of two type of raw materials (high and low value foods) sold in similar consumer-type packages. As can be seen, the energy spent in the freeze-drying process itself becomes insignificant when dealing with high-value raw materials. Freeze-drying should, therefore, not be regarded as a prohibitively expensive preservation process if it gives a reasonable added value to the product, or if it keeps its high-value, as compared to other preservation methods (Lorentzen, 1979).

Freeze-drying process consists in four main operations: freezing, vacuum, sublimation and condensing. Each of these operations shares the total energetic consumption as shown in Fig. 9. It can be noted that while sublimation takes almost half of the total energy of the process, the freezing step is not highly energy consuming. Vacuum and condensation shares are practically equal. Any new improvement to the classical vacuum freeze-drying in order to reduce energetic costs should be addressed to the following goals: (a) to improve heat transfer in order to help sublimation; (b) to cut in drying times, in order to reduce the vacuum; (c) to avoid using condensers.

Table 2
Ratio of freeze-drying to freezing costs (from Flink, 1977b and Judge, Okos, Baker, Potthast, & Hamm, 1981)

	Meat	Peas
Processing + conditioning	1.37	1.36
Processing + conditioning + storage	1.02	1.19

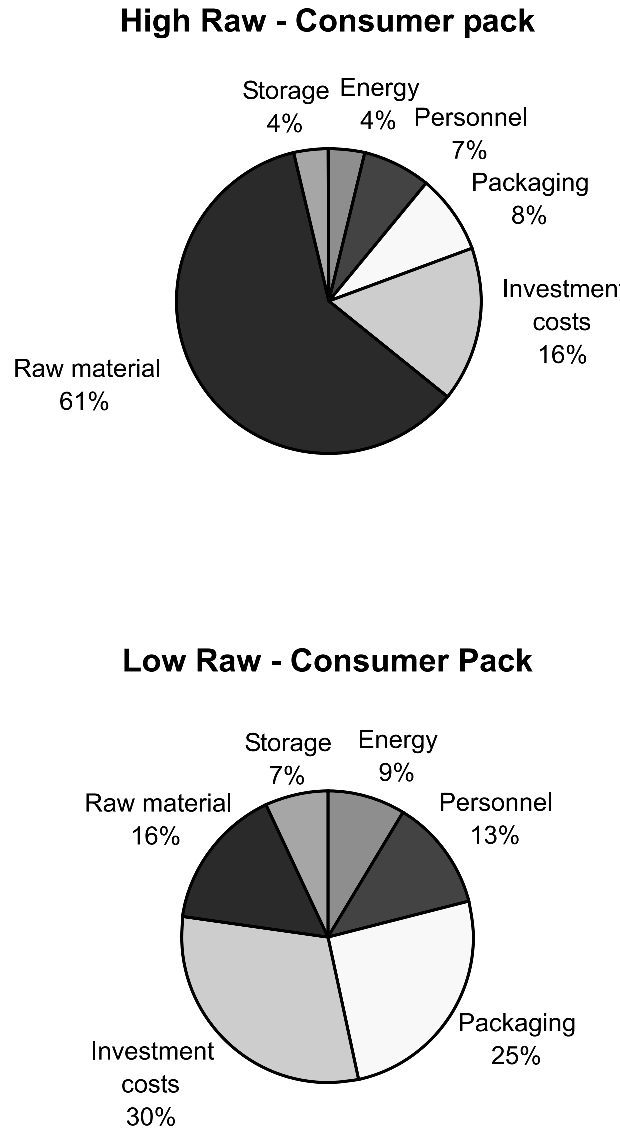

Fig. 8. Cost breakdown in two freeze-drying plants, processing high and low value foods.

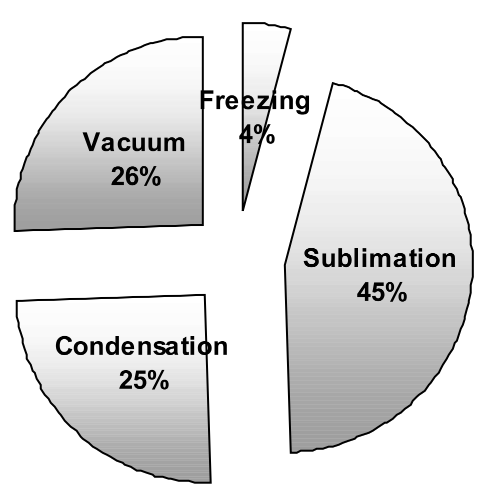

Fig. 9. Energy cost breakdown for freeze-drying process.

3.4. Technical improvements

Microwave heating provides an energy input that not only is essentially unaffected by the dry layers of the material that is undergoing freeze-drying, but is absorbed mainly in the frozen region (Sunderland, 1982b). Since the frozen region has a high thermal conductivity, microwave energy helps sublimation to decrease freeze-drying times up to 60–75% (Rosenberg & Bögl, 1987; Peltre, Arsem, & Ma, 1977). In addition, when compared to conventional freeze-drying, microwave assisted freeze-drying lead to products of similar or even higher quality (Barrett et al., 1997; Rosenberg & Bögl, 1987). Nevertheless, microwave freeze-drying is still not widely used in industry since many technical problems can be encountered (i.e., corona discharges, melting and overheating of the frozen kernel, non-uniform heating, etc.). In addition, process costs are not always lower than with conventional freeze-drying.

Adsorption freeze-drying uses a desiccant (e.g., silica gel) to create a high vapor drive at low temperatures (Bell & Mellor, 1990a). The adsorbent replaces the condenser, and let a reduction of 50% in total costs as compared to traditional freeze-drying. Despite the many advantages as compared to regular freeze-drying (Bell & Mellor, 1990b), the quality of adsorption freeze-dried foods is slightly reduced and sometimes poor as compared to that obtained by traditional freeze-drying.

Another method that has recently been developed is the fluidized atmospheric freeze-dryer. This new freeze-drying process can be defined with three words: adsorption, fluidization and atmospheric pressure (Wolff & Gibert, 1987). The cut off is approximately 34% as compared to vacuum freeze-drying (Wolff & Gibert, 1990). However, drying times are increased 1–3 times since the use of atmospheric pressure turns the control of the process from heat to mass transfer, which makes the kinetic extremely slow. Recent studies showed that in addition, quality of products is not excellent when atmospheric pressure is used instead of vacuum, since the risk of product collapse is increased (Lombraña & Villarán, 1997).

4. Conclusions

Freeze-drying process is not widely used in the food industry due to its high operation cost. Although new improvements such as adsorption, fluidization, and microwaves have been researched in the last decade in order to reduce costs, vacuum freeze-drying is, up to now, the only technology used in an industrial scale to dry coffee, spices, meats, food ingredients and other high-value foods. However, with the increasing concern about food quality, this process could be considered as a valuable alternative to preserve other foods.

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Effect of different drying methods on the phenolic, flavonoid and volatile compounds of *Stevia rebaudiana* leaves

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Abstract: Different drying methods (hot air drying, freeze drying and shade drying) were evaluated to discern the optimal conditions for the preservation of flavonoid, phenolic and volatile compounds in stevia leaves. All the methods applied affected the antioxidant and volatile compounds in dried stevia leaves differently. 2-Hexenal, hexanal and α-pinene were the most abundant volatile compounds produced by freeze drying and shade drying (21.1-19.7; 14.2-10 and 19.4-5.04 μg/g, respectively); and furan tetrahydro and α-pinene (3.2 and 3.1 μg/g, respectively) by air drying. While chlorogenic acid, coumaric acid and sinapic acid were the most abundant phenolic compounds produced by all the drying treatments (with values that ranged between 88.6-191.8; 41.7-91.3 and 33.2-178.5 mg/100g dry weight of stevia, respectively). The content of volatile compounds was higher with shade drying, whereas most flavonoids and phenolic acids had higher concentrations following freeze drying, although some flavonoids and phenolic acids exhibited a higher increment with air drying. There is no best drying treatment, however, freeze drying results in an extract with satisfactory antioxidant properties and good aromatic characteristics.

Keywords: freeze-drying; GC-MS; HPLC-DAD; shade-drying; volatile compounds

Introduction

Stevia rebaudiana is a perennial herb, native to Paraguay, which has economic value due to its high content in sweeteners.[1] In fact, its dried leaves have been used as a sweetener in South America for centuries, and nowadays extracts of steviol glycosides are consumed all over the world.[2] These extracts are 300 times sweeter than sucrose, with the advantage of having: zero calories, zero carbohydrates, and not causing spikes in blood sugar levels.[3] The European Food Safety Authority[4] recognized the safety of stevia leaf extracts for alimentary use in November 2011. Stevia leaves are more and more consumed as infusions due to their antioxidant properties, which stem from their high content in flavonoid and phenolic compounds.[5–9] In addition, their leaves have important therapeutic properties, are rich in compounds with anti-inflammatory, diuretic, anti-hypertensive, antihyperglycemic, antidiarrehal, antitumor and immunomodulatory effects.[10]

Stevia leaves, like other herbal teas or medicinal plants, need to be dried for conservation and consumption purposes. The drying process has two principal effects: preventing the growth of microorganisms and facilitating storage and transportation.[11] At the same time, drying herbs can give rise to other alterations which affect herb quality, such as changes in appearance and alterations in aroma caused by losses in volatiles or the formation of new volatiles as a result of oxidation reactions or esterification reactions.[12] Different methods can be applied to dehydrate plants. The simpler, cheaper ones include letting the leaves dry in the shade[13] or using hot air to accelerate the process.[14,15] An innovative technique using freeze drying[11] has been proven to better preserve the quality of medicinal plants.[16] It should be noted that different drying techniques influence the characteristic of the different compounds present in herbal teas. There is a great discrepancy about the extraction of active compounds from herbal teas according to the different drying techniques applied.[17] Different studies have reported changes in the antioxidant capacity of some herbal teas according to the drying method used.[11,12,15] In this line, Di Cesare *et al.*[18] and Diaz-Maroto *et al.*[19] observed changes in colour and volatile compounds of the aromatic herbs as a consequence of drying.

As far as the authors know, there is no research related to the influence of different drying methods on phenolic and volatile compounds of stevia leaves. For this reason, the aim of this study was to evaluate how the drying method (shade drying, hot air drying and freeze drying) affects phenolic and volatile compounds in stevia leaves, in order to optimize the drying method which maximizes the presence of these compounds.

Material and methods

Stevia samples and drying conditions

Organically produced *Stevia rebaudiana* Bertoni leaves from Valencia (Spain) were used in this study. Three different drying conditions were used: shade drying at 20 °C for 30 days, hot air drying at 180 °C for 3 minutes in a convective drier, and freeze drying at a vacuum pressure of 9.5×10^{-1} mm Hg for 24 hours.

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Standard compounds and reagents

HPLC-grade acetonitrile and methanol were purchased from VWR (Fontenay-sous-Bois, France), and analytical grade ethanol and ammonium acetate were purchased from Scharlab (Barcelona, Spain). The standards of apigenin, caffeic acid, catechin, chlorogenic acid, cinnamic acid, coumaric acid, 4-methoxybenzoic, 4-methylcatechol, quercetin, rutin and sinapic acid (purity > 98%) were obtained from Sigma-Aldrich (St. Louis, MO, USA). De-ionized water from MilliQ (Millipore Corp., Bedford, MA, USA) was used throughout the procedure.

Volatile compounds analysis

Volatile compounds were analysed following the method purge and trap thermal desorption described by Escriche *et al.*[20] with the only exception that 200 mg dried powder of stevia leaf and 100 μL of the internal standard 2-pentanol (10 μg/mL H_2O) were used in each analysis. This mix was shaken for several minutes to guarantee total homogenization. Samples were placed in a purging vessel flask and left in a water bath at 45 °C for 20 min. Purified nitrogen (100 mL min^{-1}) was forced through a porous frit placed at the bottom of the vessel. Volatile compounds were trapped in Tenax (TA, 20-35 mesh, Analytical Columns, New Addington, Surrey, United Kingdom), thermally desorbed (TurboMatrix TD, Perkin ElmerTM, CT,USA) and GC-MS analysed (Finnigan TRACE TM MS TermoQuest, Austin, TX, USA) using a DB-WAX capillary column (SGE, Victoria, Australia) (60 m length, 0.32 mm i.d., 1.0 μm film thickness). The analyses were carried out in triplicate.

The volatile compounds were tentatively identified using their mass spectra and their Kovats retention indices (alkanes: C8–C20 by Fluka Buchs, Schwiez, Switzerland).[20] The data were expressed in μg/g dry weight of stevia leaf, assuming a response factor equal to one.[21]

Flavonoids and phenolic acids analysis

The stevia leaves were ground in a grinding mill (A11 Basic, IKA, Staufen, Germany), and 200 mg of the dried powder were shaken in 30 mL of methanol/water (1:1 v/v) for 5 minutes. The mixture was sonicated for 10 minutes and then centrifuged at 3000 x g for 5 minutes. An aliquot of the extract was injected in the HPLC, after being filtered through filter paper (0.45 μm pore size).

Analyses of the extracts were carried out using HPLC-Alliance 2695, with a 2996 photodiode array detector (Waters, Milford, Massachusetts, USA). Flavonoids and phenolic compounds were separated on a Brisa LC2, C18 column (250 x 4.6mm x 5 μm) (Teknokroma, Barcelona, Spain). The binary mobile phase consisted of solvent A (ACN) and solvent B (water and formic acid, 99:1). Binary gradient conditions were used: initial, 90% B, linear gradient to 40% B at 25 min and then to 20% B at 26 min; holding until 30 min; followed by a linear gradient to initial condition at 35 min and a final hold at this composition until 40 min. The column was maintained at 30 °C. The flow-rate and the injection volume were 0.5 mL/min. and 10 μL, respectively.

Chromatograms were recorded at three wavelengths (290, 320 and 360 nm). Flavonoids and phenolic acids were identified by comparison of chromatographic retention times and UV spectral characteristics of unknown analytes with authentic standards. Calibration curves were constructed via least squares linear regression analyses of the ratio of the peak area of each representative compound versus the respective concentration. Quantitative results were expressed as mg of component per 100 g dry weight of stevia.

The pure standard of flavonoids and phenolic acids were diluted with methanol to obtain a final concentration of 1 mg/mL for the stock standard solution. The working standard solution was obtained at a concentration of 100 ng/mL in water. The stock standard solution was stored at −20 °C and the working standard solution at +4 °C.

Calibration curves obtained from standard solutions (0.5–10 ng/mL) were used to perform the quantification. Samples were spiked to verify the absence of a matrix effect in the analysis. An internal quality control (a standard solution) was injected into the equipment as a first step, before each batch of the sample, in order to ensure the quality of the results and evaluate the stability of the proposed method.

Validation of flavonoids and phenolic acids analysis method

The guidelines established by the EU Commission Decision[22] were followed in order to validate the analytical methodology

Table 1. Mean and standard deviation of flavonoid and phenolic compounds quantified in the three drying methods (mg/100 g dry weight of stevia leaf)

mg/100 g stevia leaf	Freeze drying	Air drying	Shade drying	Anova F-ratio
apigenin	0.24(0.04)^a	0.25(0.02)^a	0.39(0.02)^a	1^{ns}
caffeic acid	1.22(0.02)^b	0.71(0.04)^a	0.75(0.03)^a	350***
catechin	8.35(0.38)^c	6.18(0.33)^b	4.38(0.42)^a	55**
chlorogenic acid	191.84(0.7)^c	167.56(0.12)^b	88.60(3.19)^a	1621***
cinnamic acid	0.27(0.07)^{ab}	0.34(0.02)^b	0.19(0.02)^a	7^{ns}
coumaric acid	91.35(0.16)^c	70.36(0.30)^b	41.71(0.48)^a	10616***
4-methoxybenzoic	7.48(0.39)^a	26.28(0.43)^c	15.39(0.2)^b	1394***
4-methylcatechol	2.49(0.02)^b	2.99(0.56)^b	0.73(0.07)^a	26*
quercetin	0.33(0.03)^a	0.28(0.02)^a	0.39(0.06)^a	5^{ns}
rutin	20.07(0.13)^c	15.08(0.22)^b	7.05(0.02)^a	4174***
sinapic acid	178.56(0.7)^c	165.14(1.53)^b	33.21(0.23)^a	13544***

a, b, c: ANOVA homogenous groups.
* $p<0.05$.
** $p<0.01$.
*** $p<0.001$, ns: non significant.

employed to analyse the flavonoids and phenolic acids. For this purpose, several parameters were studied: linearity, accuracy and precision (repeatability and reproducibility). The accuracy of the method was established through recovery studies and the precision was verified by repeatability (intraday precision) and reproducibility (interday precision).

Statistical analysis

An analysis of variance (ANOVA) (α = 0.05) with least significant difference (LSD) test using Statgraphics Plus 5.1 was performed on the data from flavonoids and phenolic acids as well as the volatile compounds. In addition to this, the data were analysed using multivariate techniques, applying the software Unscrambler version 9.7 (CAMO, 2005). The variables were weighted with the inverse of the standard deviation of all objects in order to compensate for the different scales of the variables. A Principal Components Analysis (PCA) was applied to describe the relationship between the flavonoids and phenolic compounds together with the volatile profile.

Results and discussion

Influence of drying method on the phenolic and flavonoid compounds

The average value of phenolic compounds (mg /100 g dry weight of stevia) quantified in the stevia leaves obtained using different

Table 2. Semiquantification of volatile compounds (μg/g dry weight of stevia assuming a response factor equal to 1) in stevia dried leaves (n = 3)

Volatile compounds	Shade drying	Air drying	Freeze drying	KI cal	ID	Anova F-ratio
Alcohols						
1-penten-3-ol	0.60(0.09)^a	0.15(0.01)^a	1.96(0.02)^b	774	MS;KI	14.8*
1-pentanol	0.42(0.01)^a	0.63(0.03)^a	0.33(0.04)^a	704	MS;KI	2.43^{ns}
1-octen-3-ol	5.85(0.08)^c	0.68(0.02)^a	2.37(0.08)^b	980	MS;KI	149***
3,7dimethyl-1,3 octadien-3-ol	5.50(0.07)^c	0.93(0.01)^a	2.35(0.10)^b	1110	MS;KI	99***
2 ethyl-1-hexanol,	0.88(0.02)^b	0.18(0.01)^a	0.27(0.02)^a	1028	MS;KI	46**
Aldehydes						
2-ethyl-butanal	2.64(0.12)^b	0.29(0.02)^a	0.33(0.05)^a	662	MS;KI	23.7**
3-methyl-butanal	3.01(0.20)^b	0.11(0.01)^a	0.78(0.06)^a	676	MS;KI	10.87*
pentanal	2.25(0.17)^b	1.26(0.02)^a	1.22(0.09)^a	780	MS;KI	2.03*
hexanal	14.23(0.53)^b	0.86(0.01)^a	10.02(0.26)^b	860	MS;KI	27.4**
heptanal	0.41(0.02)^b	0.11(0.01)^a	0.08(0.01)^a	896	MS;KI	14.3*
2-hexenal	21.09(0.66)^b	0.83(0.08)^a	19.78(0.96)^b	768	MS;KI	24.9**
2-heptenal	2.63(0.02)^b	0.28(0.01)^a	0.64(0.07)^a	932	MS;KI	121***
2-4 heptadienal	3.29(0.05)^c	0.36(0.02)^a	1.69(0.08)^b	1015	MS;KI	75***
octanal	0.34(0.01)^a	0.20(0.01)^a	0.29(0.02)^a	1004	MS;KI	3.7^{ns}
nonanal	2.34(0.07)^a	1.28(0.08)^a	1.73(0.13)^a	1106	MS;KI	3.5^{ns}
decanal	0.68(0.02)^a	0.74(0.01)^a	1.06(0.08)^a	1204	MS;KI	1.9^{ns}
Hydrocarbons						
benzene	0.44(0.07)^a	0.19(0.03)^a	0.12(0.01)^a	662	MS;KI	1.15^{ns}
1-heptene	1.92(0.08)^b	0.05(0.01)^a	0.81(0.05)^a	690	MS;KI	21.08**
1-octene	2.37(0.12)^b	0.11(0.01)^a	1.06(0.14)^{ab}	790	MS;KI	11.12*
Ketones						
3-buten-2-one	0.50(0.01)^a	0.69(0.11)^a	0.49(0.05)^a	707	MS;KI	0.2^{ns}
4-hydroxy-2-butanone	0.74(0.07)^a	0.58(0.06)^a	0.48(0.04)^a	720	MS;KI	0.4^{ns}
6 methyl-5-hepten-2-one	0.29(0.01)^a	0.22(0.02)^a	0.29(0.03)^a	987	MS;KI	0.4^{ns}
Terpenes						
α-pinene	19.40(0.70)^b	3.14(0.10)^a	5.04(0.52)^a	912	MS;KI	25.7**
limonene	0.72(0.01)^a	0.54(0.02)^a	0.45(0.05)^a	1024	MS;KI	2.2^{ns}
caryophyllene	8.24(0.08)^b	1.68(0.15)^a	2.36(0.28)^a	1432	MS;KI	47**
Nitriles						
2-hydroxy-2-methyl-propanenitrile	3.41(0.17)^b	0.56(0.02)^a	0.53(0.03)^a	752	MS;KI	19.5**
Furanes						
tetrahydro furan	2.61(0.31)^a	3.25(0.15)^a	1.34(0.16)^b	628	MS;KI	1.26*
Sulfur compounds						
dimethyl sulfide	1.02(0.01)^b	0.18(0.01)^a	0.39(0.04)^a	741	MS;KI	41.36**

a, b, c: ANOVA homogenous groups.
* $p<0.05$.
** $p<0.01$.
*** $p<0.001$, ns: non significant.
KI cal: Kovats retention indices calculated.
ID: method of identification, MS (comparison with mass spectrum from NIST library) and KI (comparison of Kovats index with the literature).[20]

drying methods (shade drying, freeze drying and air drying), as well as the ANOVA F-ratio and homogenous groups for each of the analysed compounds are shown in Table 1. 11 compounds were identified in all samples: apigenin, caffeic acid, catechin, chlorogenic acid, cinnamic acid, coumaric acid, 4-methoxybenzoic, 4-methylcatechol, quercetin, rutin and sinapic acid.

With regard to the validation parameters, good linearity was obtained, with R^2 values ranging from 0.991 for 4-methoxybenzoic to 0.999 for quercetin, catechin and 4-methylcatechol. The range of the average recoveries varied from 90% for caffeic acid to 117% for sinapic acid. The RSD_r (repeatability standard deviation) for all compounds was less than 9% and the RSD_R (reproducibility standard deviation) was always less than 13%. In both cases the values were below 20%, and therefore in agreement with the requirements of the Commission Decision.[22]

The highest F-ratio in Table 1 shows that coumaric and sinapic acid were most influenced by the drying method. The concentrations of other compounds such as apigenin, quercetin and cinnamic acid showed practically no differences as a result of applying the three treatments.

The majority of the compounds analysed reached their maximum values with the freeze drying method. Compounds such as chlorogenic acid, coumaric acid and sinapic acid exhibited a higher concentration after freeze drying (191.84, 91.35 and 178.56 mg/100 g stevia leaf, respectively) and air drying (167.56, 70.36 and 165.14 mg/100 g stevia leaf, respectively) than shade drying (88.60, 41.71 and 33.21 mg/100 g stevia leaf, respectively). However, the values obtained for 4-metoxybenzoic following freeze drying (7.48 mg/100 g stevia leaf) were lower than those for the other treatments (air drying-26.28 mg/100 g stevia leaf and shade drying-15.39 mg/100 g stevia leaf).

Many antioxidant compounds have been identified in stevia leaves by different authors, but their conclusions with respect to both the specific compounds and the concentration levels are very different and even contradictory. This can be explained by the fact that the drying methods employed were different in each case. However, in some studies it was not even mentioned. Different flavonoids (flavonols and flavones) have been identified: quercetin and its derivatives, apigenin and its derivatives, kaempferol-3-O-rhamnoside, luteolin and their derivatives[23–25] in stevia dried leaves. Karaköse *et al.*[26] identified 24 chlorogenic acids using LC-ESI-MS. Muanda *et al.*[8] identified (at room temperature) the same phenolic and flavonoid compounds in stevia dried leaves as in the present work, with the exception of 4-methoxybenzoic acid, 4-methylcatechol and sinapic acid. Kim *et al.*[27] identified six phenolic acids: pyrogallol, 4-methoxybenzoic acid, 4-methylcatechol, sinapic acid, coumaric acid and cinnamic acid (at 40 °C for 12 h). All of them were identified in the present study, with the exception of pyrogallol. It is important to highlight that the values obtained by Kim *et al.*[27] were lower than those reported by Muanda *et al.*[8]

Considering other medicinal herbal teas, Lin *et al.*[11] claimed that freeze drying was the best method for preserving the higher contents of caffeic acid derivatives and total phenolics in *Echinacea Purpurea* leaves. Ferreira and Luthria[28] obtained lower levels of antioxidant capacity for shade drying than hot air drying in *Artemisia annua* L. leaves.

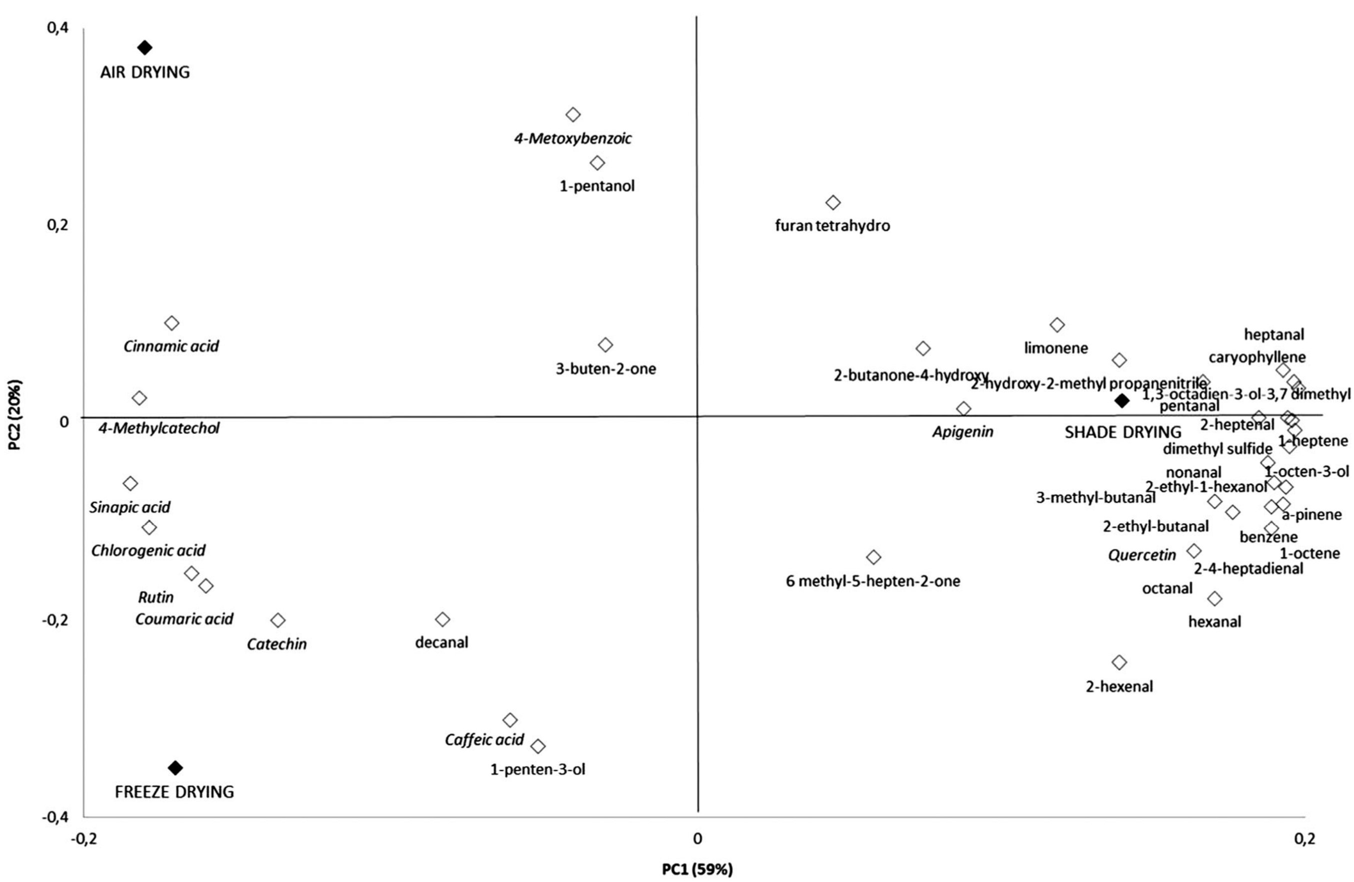

Figure 1. Bi-plot of Principal Components Analysis for the drying treatments (black diamond ♦) and the analysed variables: phenolic, flavonoid and volatile compounds (white diamond ◊)

Influence of drying method on the volatile compounds

30 volatile compounds were tentatively identified. Table 2 shows the mean concentration values of the quantified volatile compounds (expressed as μg/g dry weight of stevia leaf) as well as their standard deviations (SD) for the three drying methods.

The most abundant compounds produced by shade drying and freeze drying were 2-hexenal (21.09 and 19.78 μg/g), hexanal (14.23 and 10.02 μg/g) and α-pinene (19.40 and 5.04 μg/g), respectively. The most abundant compounds produced by air drying, were furan tetrahydro (3.25 μg/g) and α-pinene (3.14 μg/g).

In contrast to the phenolic and flavonoid compounds, shade drying better preserves the volatile fraction of stevia leaves in comparison with freeze drying and air drying.

There are a few studies about the volatile fraction of stevia leaves and all of them analysed the volatile compounds in the essential oils in stevia. Muanda *et al.*[8] identified 34 volatile compounds, Moussa *et al.*[29] found 22 compounds, Turko *et al.*[30] reported 23 compounds and Zygadlo *et al.*[31] identified 41 compounds, only five of them (α-pinene, hexanal, limonene, 1-octen-3-ol, caryophyllene) were identified in this study, which is logical because in the present work the analysis was performed directly on the stevia dried leaves and not on the essential oil.

Global behavior of phenolic and volatile compounds

A PCA was applied in order to appreciate the overall effect that the drying method has on phenolic and volatile compounds together. The corresponding bi-plot obtained (scores ‘treatments’ and loading ‘variables’) is shown in Figure 1 (PC1 explained 59% of the total variance and PC2, 20%). The proximity between variables indicates the correlation between them, and in the case of drying treatments similar behaviour. In general, this figure shows opposing behaviour between the two groups of variables (phenols and volatiles) with respect to the effect of the drying treatments applied.

The shade drying treatment is placed at the far end of the right axis in the figure, which corresponds to the highest values of the volatile compounds and the lowest of the phenolic compounds. On the contrary, freeze drying and air drying are placed on the opposite side (left axis), which corresponds to the highest content of phenolic compounds. The only exceptions to this general pattern are apigenin and quercetin which are placed with the volatile compounds even though they are antioxidant compounds.

Apparently, some volatile compounds could be generated as a result of oxidation and degradation reactions involving the phenolic and acid compounds,[32] so perhaps freeze drying helps to preserve them, whereas drying in the shade favours degradation processes.

Conclusions

All the drying methods applied (freeze drying, shade drying and air drying) affected the antioxidant and volatile compounds in the dried stevia leaves. The two types of compounds reacted differently; the content of volatile compounds was higher with shade drying whereas most flavonoids and phenolic acids had higher concentrations when freeze drying was applied. However, some flavonoids and phenolic acids exhibited a higher increment with air drying. Therefore there is no ideal drying treatment which can be chosen, although freeze drying is the most recommendable if an extract with sufficient antioxidant properties and satisfactory aromatic characteristics is desired.

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Effect of pre-treatment conditions and freeze-drying temperature on the process kinetics and physicochemical properties of pepper

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ABSTRACT

This study shows the effects of blanching, citric acid addition, and drying temperature on the freeze-drying kinetics, L-ascorbic acid content, colour, and antioxidant activity of freeze-dried pepper. The process was performed at 20 °C, 40 °C, and 60 °C and with a constant pressure in a drying chamber at 63 Pa. The samples of pepper were pulped before drying. Blanching of pepper reduced the drying time to approximately 30%. The shortest drying time (about 290 min) was found for blanched pepper that was freeze-dried at 60 °C, whereas the samples of pepper freeze-dried at 20 °C and without blanching required the longest drying time (about 900 min). The kinetics of freeze-drying of pepper pulp are best described by using the Page model. The addition of citric acid increased the redness and yellowness of dried pepper, whereas an increase in drying temperature caused a decrease in the total phenolics content, antioxidant activity, and colour coordinates of all samples. The highest L-ascorbic acid content was found in unblanched pepper and when the temperature of drying did not exceed 40 °C. Water blanching pretreatment had the most negative effect on total phenolics content and antioxidant activity of dried pepper.

1. Introduction

The pepper (*Capsicum annuum*, L.), indigenous to South and Central America, is now grown worldwide and has been incorporated into many different cuisines (Vega-Gálvez et al., 2009). The dehydrated and powdered fruits of red pepper, called paprika, are most widely used as a food colorant (Topuz, Feng, & Kushad, 2009). Moreover, this spice can modifying the flavour of food, due to its characteristic taste and pungency (Martín et al., 2017). Fruits of pepper can vary tremendously in colour, shape, and size, both between and within the species. Ripe pepper fruits belonging to different varieties display a range of colours, from white to deep red (Arimboor, Natarajan, Menon, Chandrasekhar, & Moorkoth, 2015). The colour and degree of pungency are valued as major quality parameters in the paprika trade. There are many nutraceutical benefits associated with the consumption of pepper. Especially, red pepper has been recognised as an excellent source of different phytochemicals such as flavonoids, quercetin, vitamin C and capsaicinoids (Vega-Gálvez et al., 2009). Furthermore, red pepper fruits contain more than 20 different carotenoids, which are the primary source of their colours (Hallmann & Rembiałkowska, 2012). Carotenoids are lipophilic yellow-orange-red pigments found in photosynthetic plants, algae, and microorganisms. These compounds are commercially used as food and feed additives and are also used in pharmaceutical, nutraceutical, and cosmeceutical products (Arimboor et al., 2015). Red pepper contains mainly such carotenoids as β-carotene, lutein, and capsanthin (Rodriguez-Amaya, Kimura, Godoy, & Amaya-Farfan, 2008), which are source of natural colours in plants. The antioxidant potential of carotenoids is of particular significance to human health. Data obtained from epidemiological studies and clinical trials strongly support the observation that adequate carotenoid supplementation may significantly reduce the risk of several disorders that are mediated by reactive oxygen species (Fiedor & Burda, 2014). Besides, pepper is a richer source of vitamin C, more than other vegetables and fruits that are commonly recognised as a source of this vitamin (Dürüst, Sümengen, & Dürüst, 1997).

Traditionally, paprika is obtained by sun drying red peppers (Condori, Echazu, & Saravia, 2001; Topuz et al., 2009). Magied and Ali (2017) found that the solar drying method yielded high values for colour and rehydration ratio of dried pepper than the conventional drying method. However, solar drying is a long process and takes up to 10 days (Oberoi, Ku, Kaur, & Baboo, 2005); moreover, in many countries, weather conditions are inadequate for such drying. This technique

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also increases the possibility of fungal proliferation, which creates favourable conditions for mycotoxin contaminations (Arslan & Ozcan, 2011). Thus, hot-air drying is often used for pepper dehydration with a drying temperature of 50–60 °C. This method reduces the drying time to a maximum of 20 h (Minguez-Mosquera, Jaren-Galan, & Garrido-Fernandez, 1994). Unfortunately, this dehydration method negatively affects the quality attributes of paprika such as texture, flavour, colour, and nutritional value (Topuz et al., 2009). Park and Kim (2007) compared the different methods of pepper drying; they found that freeze-drying is the most suitable dehydration method for stabilizing the antioxidant compounds and red pigment of pepper. Data obtained from several other authors have also revealed that freeze-drying is one of the best food preservation methods. Compared to others food drying techniques, the most significant benefits of freeze-drying include the following: retention of morphological, biochemical, and immunological properties, high viability, long shelf life, retention of structure, and high recovery of violates (Ciurzyńska & Lenart, 2011). Especially, freeze-drying in comparison with hot-air drying results in products with higher content of vitamins and antioxidants, better colour, crisper texture, and better rehydration capacity (Orak et al., 2012:; Lee, Oh, Han, & Lim, 2012; Sogi, Siddiq, Greiby, & Dolan, 2013). Unfortunately, this method of drying is time-consuming and expensive. This process can by shortened by using different drying conditions and adequate methods of raw material pretreatment (Rudy et al., 2015). Moreover, the different methods of food pretreatments before freeze-drying have a definite effect on the quality attribute of the final products (Ciurzyńska, Lenart, & Gręda, 2014; Dehghannya, Gorbani, & Ghanbarzadeh, 2017; Garcia-Noguera, Oliveira, Weller, Rodrigues, & Fernandes, 2014; Prosapio & Norton, 2017). To the best of our knowledge, these aspects have not been studied for freeze-dried pepper. Thus, the aim of this study was to determine the effect of pepper pretreatments and freeze-drying temperature on the process kinetics and physicochemical properties of dried pepper.

2. Materials and methods

2.1. Materials

Mature pods of red pepper (*Capsicum annuum*, L., cv. Kaskada) were grown and harvested in September 2017 in Lublin, Poland. The pods were stored at 4 °C before processing for a maximum period of six days. The samples of pepper were selected visually based on colour and freshness, and with no sign of mechanical damage. Next the pods were washed using tap water, cut into 1.0-cm thick slabs lengthwise, the inside seeds were removed, and the water content was determined (AOAC, 1990).

2.2. Pepper pretreatments

The samples of pepper were prepared for freeze-drying as follows:

- pepper was blanched in hot water (WB) (90 °C) for 1 min using a thermostated water bath (Horyzont UL-1, Poland),
- samples of pepper were placed in the microwave oven (MB) (Daewoo KOR-GL05, China) for 1.5 min at a power of 650 W (Castro et al., 2008; Dorantes-Alvareza, Jaramillo-Floresa, González, Martinez, & Parada, 2011).

After blanching, all samples were cooled at room temperature and the surface water was removed using absorbent paper. Blanched and control samples of pepper were dried after blending into a homogenous mass (30 s) using a knife blender (Braun, Model, 2001; Germany). Subsequently, citric acid (CA) (250 mg/100 fresh pepper) was to the pepper purée. In addition, a control sample (pepper purée with no acid added) was freeze dried as well.

2.3. Freeze-drying method

Samples of raw peppers (a single layer of about a 5 mm layer of pepper purée) were placed on a stainless steel plated with a diameter of 21 cm and frozen at −25 °C for 48 h using a freezer (Liebherr GTL-4905, Germany). The same sample was freeze dried each time using an ALPHA 1–4 laboratory freeze dryer according to the method described by Dziki et al. (2018). The process of drying was performed at 20 °C, 40 °C, and 60 °C and with a constant pressure of 63 Pa in the drying chamber until the sample reached an equilibrium moisture of about 70 g H_2O/kg fresh weight (FW). After freeze-drying, the material was stored in a dark place and in tightly closed polyethylene bags at a temperature of 20–22 °C.

2.4. Characteristics of drying curves

Based on the measurements of mass loss taken over the course of the experiment, drying curves were obtained as functions of water ratio (MR) versus time, using the following equation (Motevali, Younji, Chayjan, Aghilinategh, & Banakar, 2013):

$$MR = \frac{u}{u_0}, \tag{1}$$

where u is the water content during drying [kg H_2O/kg dry matter (DM)] and u_0 is the initial water content [kg H_2O/kg DM]. For modelling, the freeze-drying equations in Table 1 were tested to select the best model for describing the drying curve of peppers.

2.5. Sample preparation

The individual dried pepper samples were ground using the laboratory knife mill (Grindomix GM 200, Retsch, Dusseldorf, Germany). The powdered samples of paprika (particles < 0.2 mm) were then subjected to other tests.

2.6. Colour evaluation

The colour of dried pepper was measured using a CR-400 Chromameter (Minolta). The analyses of the colour values were performed three times with each dried pepper sample. Three parameters, L* (lightness), a* (redness), and b* (yellowness), were used to study the colour changes. From the data obtained, colour intensity (Δ*E*) was

Table 1
Equations applied to drying curves.

Model number	Model name	Model equation	References
1	Henderson and Pabis	$MR = a \cdot \exp(-k \cdot \tau)$	Henderson and Pabis (1961)
2	Logarithmic	$MR = a \cdot \exp(-k \cdot \tau) + b$	Sarimeseli (2011)
3	Newton	$MR = \exp(-k \cdot \tau)$	El-Beltagy, Gamea, and Amer Essa (2007)
4	Page	$MR = \exp(-k \cdot \tau^n)$	Diamante and Munro (1993)
5	Two term	$MR = a \cdot \exp(-k \cdot \tau) + b \cdot \exp(-k_i \cdot \tau)$	Arslan and Ozcan (2008)
6	Wang and Singh	$MR = 1 + a \cdot \tau + b \cdot \tau^2$	Wang and Singh (1978)

k, k_i – drying coefficients [min^{-1}]; a, b – coefficients of the equations; n – exponent; τ – time [min].

calculated (Jokić et al., 2009).

2.7. L-ascorbic acid determination

The content of L-ascorbic acid in the fresh and dried pepper was determined using Ultra-performance Liquid Chromatography (UPLC) (Spínola, Mendes, Câmara & Castilho, 2012). To prepare the extract for analysis, 0.5 g of fresh and pulped pepper or dried pepper was added to 25 mL of extraction solution (3% MPA – 8% acetic acid – 1 mM EDTA), vortexed in the darkness, and then centrifuged for 10 min at a velocity of 6000 rpm. The supernatants were filtered through 0.22-µm PTFE filters (Milipore, USA). An Acquity UPLC system (Waters Corporation, USA) with a Waters Acquity UPLC photodiode array (PDA) detection system and EmpowerTM software (Waters Corporation, USA) were used to record detection signal and process the peak areas. The L-ascorbic acid in samples was determined by comparison with the retention time of standard and match with the UV absorption spectrum according to the procedure described by Nowacka et al. (2018).

2.8. Determination of total phenolics and antioxidant activity

To prepare the extract for analysis, 0.5 g samples of fresh or dried pepper (0.5 g) were extracted for 30 min with 10 mL of 80% ethanol (v/v). The extracts were separated via decantation and the residues were extracted again with 10 mL of 50% ethanol (v/v). Extracts were combined and stored in darkness at −20 °C.

The total phenolics content (TPC) was estimated according to the Folin–Ciocalteau method (Singleton & Rossi, 1965). The amount of total phenolics was expressed as gallic acid equivalents (GAE).

Antiradical activity (AA) was determined using an improved ABTS decolorization assay (Re et al., 1999). Chelating power (CHEL) was studied using the method of Guo, Lee, Chiang, Lin, and Chang (2001). Reducing power (RED) was determined using the method described by Oyaizu (1986). All activities were evaluated in triplicate and expressed as EC_{50} – extract concentration provided 50% of activity based on the dose-dependent mode of action. EC_{50} value (mg DM/mL) is the effective concentration at which the absorbance was 0.5 for reducing power and was obtained through interpolation from linear regression analysis.

2.9. Statistical analysis

Data represent the mean and standard deviation from three independent dryings. Measurement scores were subjected to an analysis of variance (ANOVA). When significant differences in ANOVA were detected, the means were compared using Tukey's test. Statistical analysis was performed at a significance level of a $\alpha = 0.05$ using Statistica 13.0 by Statsoft. A regression analysis was also performed. The coefficient of determination R^2, root mean square error (RMSE), and the reduced χ^2 values were calculated (Akpinar, Midilli, & Bicer, 2003).

3. Results and discussion

3.1. Drying kinetics

The drying curves of pepper are seen in Fig. 1. The initial moisture content of the paprika was 914 g H_2O/kg fresh weight. The addition of CA had no significant effect on the drying curves. Thus, the average values of *MR* for individual temperatures were obtained. The results showed that both WB and MB reduced the drying time. The shortest drying time (about 290 min) was found for pepper that was blanched using MB and WB and freeze-dried at 60 °C, whereas the pepper without pretreatments and dried at same temperature required about 80 min longer drying time. The samples of pepper freeze-dried at 20 °C and without blanching required the longest drying time (about 900 min). In this instance blanching reduced drying time by about 250 min. Wang et al. (2017a) showed that blanching of red pepper before air drying

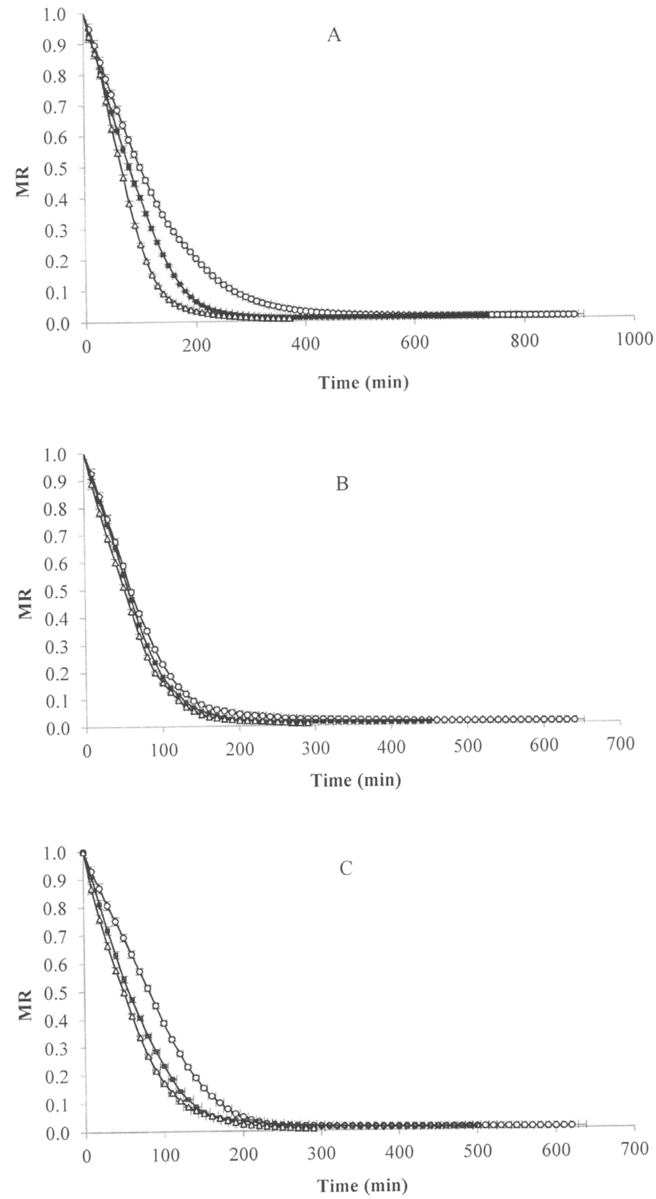

Fig. 1. Drying curves of freeze-dried pepper: A - with and without citric acid, B - microwave blanching, C - water blanching; MR - water ratio, ○– 20 °C, ■– 40 °C, Δ– 60 °C.

significantly reduced the drying time. Further, an increase of freeze-drying temperature from 20 °C to 60 °C decreased the drying time about two-fold. This tendency was found both for control and blanched samples. Our previous study also showed similar relation between drying time of cranberries and temperature of heating plates during lyophilization (Rudy et al., 2015). The results of regression analyses for the models that were used to describe the freeze drying kinetics of the control and blanched peppers are presented in Tables 2 and 3. For almost each of the models, a good fit for the experimental data was observed. Only in the case of the model used by Wang and Singh, where the temperature of the heating plates was 20 °C and 40 °C, this fit was noticeably lower and the lowest values of R^2 and the highest values of RMSE were obtained. Based on the results of regression Page model seems to be the best suited to describe the freeze-drying process of pepper. This model is often used as the best equation to represent the drying kinetics for the freeze-drying and hot air drying of fruits and vegetables (Evin, 2011; Marques & Freire, 2005).

Table 2
Statistical analysis of models describing kinetics of freeze drying of pepper at 20 °C.

Model name	DT (°C)	Sample								
		FD			WB			MB		
		R^2	*RMSE*	χ^2	R^2	*RMSE*	χ^2	R^2	*RMSE*	χ^2
Henderson and Pabis	20	0.99618	0.00222	$5.07\ 10^{-6}$	0.98127	0.01057	0.00011	0.98902	0.00503	$2.61\ 10^{-5}$
	40	0.98687	0.00966	$9.83\ 10^{-5}$	0.99199	0.00369	$1.42\ 10^{-5}$	0.98625	0.00662	$4.57\ 10^{-5}$
	60	0.97933	0.01140	0.00013	0.99392	0.00267	$7.66\ 10^{-6}$	0.98824	0.00549	$3.23\ 10^{-5}$
Logarithmic	20	0.99618	0.00222	$5.11\ 10^{-6}$	0.98206	0.01013	0.00011	0.98930	0.00490	$2.52\ 10^{-5}$
	40	0.98652	0.006985	$5.08\ 10^{-5}$	0.99201	0.00369	$1.44\ 10^{-5}$	0.98652	0.00649	$4.50\ 10^{-5}$
	60	0.98186	0.01001	0.00011	0.99517	0.00212	$5.01\ 10^{-6}$	0.99028	0.00453	$2.28\ 10^{-5}$
Newton	20	0.99324	0.003938	$1.56\ 10^{-5}$	0.97375	0.01483	0.00022	0.98375	0.00745	$5.63\ 10^{-5}$
	40	0.98101	0.01027	0.00010	0.98846	0.00532	$2.89\ 10^{-5}$	0.98000	0.00963	$9.48\ 10^{-5}$
	60	0.96898	0.01711	0.00030	0.99209	0.00348	$1.25\ 10^{-5}$	0.98381	0.00755	$5.9\ 10^{-5}$
Page	20	0.99919	0.000468	$2.24\ 10^{-7}$	0.99731	0.00152	$2.38\ 10^{-6}$	0.99622	0.00173	$3.09\ 10^{-6}$
	40	0.99796	0.00149	$1.24\ 10^{-6}$	0.99793	0.00095	$9.48\ 10^{-7}$	0.99818	0.00087	$7.97\ 10^{-7}$
	60	0.99828	0.00094	$9.48\ 10^{-7}$	0.99862	0.00061	$3.92\ 10^{-7}$	0.99836	0.00076	$6.26\ 10^{-7}$
Two-factor	20	0.99618	0.00223	$5.19\ 10^{-6}$	0.98128	0.01058	0.00012	0.98902	0.00503	$2.69\ 10^{-5}$
	40	0.98687	0.00711	$5.33\ 10^{-5}$	0.99327	0.00310	$1.04\ 10^{-5}$	0.98893	0.00532	$3.11\ 10^{-5}$
	60	0.97933	0.01140	0.00014	0.99392	0.00267	$8.25\ 10^{-6}$	0.98824	0.00549	$3.47\ 10^{-5}$
Wang and Singh	20	0.81165	0.10986	0.01234	0.85045	0.08447	0.00737	0.62518	0.17177	0.03044
	40	0.76205	0.12876	0.01704	0.79490	0.09462	0.00932	0.83881	0.07759	0.00629
	60	0.96037	0.02186	0.00051	0.96019	0.01751	0.00033	0.96622	0.01576	0.00026

DT – drying temperature, FD – freeze drying, WB - water blanching, MB – microwave blanching.

Table 3
Coefficient values in the Page model describing the freeze drying of pepper.

DT (°C)	Sample	Parameter	
		k	n
20	FD	0.0030	1.185
40		0.0018	1.370
60		0.0013	1.510
20	WB	0.0012	1.461
40		0.0044	1.261
60		0.0072	1.186
20	MB	0.0030	1.335
40		0.0026	1.399
60		0.0043	1.310

DT – drying temperature, FD – freeze drying, WB - water blanching, MB – microwave blanching.

3.2. Colour assessment

Table 4 presents the results for the colour attributes for dried pepper. The average values for the colour parameters of fresh pepper were: L* = 33.1, a* = 28.1, b* = 19.7. The freeze-drying of pepper significantly increased the lightness and yellowness of dried fruits, whereas redness increased when the temperature was 40 °C and 60 °C and decreased when the pepper was lyophilized at 20 °C. This tendency was obtained for all samples of dried peppers. The highest values of colour coordinates and colour intensity where obtained for samples dried at 40 °C. The decreasing of colour coordinated at 20 °C it is probably caused by longer time of drying and degradation of carotenoids. Similar tendency was found when pepper was freeze-dried at 60 °C. Colour is the most studied parameter of dried pepper; commercially, the red colour intensity is the main quality criterion. The colour intensity of the final product depends on the variety of pepper (Gómez, Pardo, Navarro & Varon, 1998) but is also highly influenced by the drying method (Topuz et al., 2009). Park and Kim (2007) showed that lyophilization is the most suitable drying method for maintaining the

Table 4
Effect of freeze drying temperature and pre-treatment on colour parameters of pepper.

Sample	DT (°C)	DC			
		*L**	*a**	*b**	*ΔE*
FP	–	33.1 ± 1.52[a]	28.1 ± 0.73[c]	19.7 ± 0.23[i]	-
FD	20	39.2 ± 0.26[def]	34.8 ± 0.43[f]	27.0 ± 0.35[ac]	11.3 ± 0.63[de]
	40	41.1 ± 0.58[h]	37.2 ± 0.22[g]	28.8 ± 0.45[bd]	14.8 ± 0.56[f]
	60	35.5 ± 0.72[b]	27.6 ± 0.36[c]	23.8 ± 0.49[g]	4.9 ± 0.33[a]
WB	20	40.6 ± 0.38[gh]	30.9 ± 0.39[d]	28.6 ± 0.45[b]	11.8 ± 0.36[e]
	40	43.0 ± 0.44[i]	35.6 ± 0.35[a]	32.6 ± 0.47[f]	17.7 ± 0.58[h]
	60	38.9 ± 0.47[cde]	25.9 ± 0.60[b]	27.4 ± 0.40[a]	10.0 ± 0.27[c]
MB	20	39.8 ± 0.31[efg]	32.4 ± 0.49[e]	27.9 ± 0.38[ab]	11.2 ± 0.43[d]
	40	42.4 ± 0.67[i]	36.1 ± 0.34[a]	29.7 ± 0.53[de]	15.5 ± 0.47[fg]
	60	38.1 ± 0.50[c]	26.2 ± 0.32[b]	25.1 ± 0.43[h]	7.8 ± 0.31[b]
CA	20	40.1 ± 0.59[fgh]	39.3 ± 0.25[h]	30.3 ± 0.56[e]	16.5 ± 0.58[gh]
	40	43.2 ± 0.45[i]	42.8 ± 0.23[i]	32.2 ± 0.23[f]	21.4 ± 0.71[i]
	60	38.6 ± 0.49[cd]	36.1 ± 0.28[a]	26.4 ± 0.50[c]	11.4 ± 0.42[de]

*The values are expressed as mean ± SD (n = 3); DT – drying temperature, FP - FP – fresh pepper, DC – dimension of colour, FD – freeze dried (without pretreatment), WB – water blanching, MB – microwave blanching, CA – with citric acid addition, The values designated by the different small letters in the columns of the table are significantly different (α = 0.05).

Table 5
Ascorbic acid content, total phenolics content and antioxidant activity of dried pepper.

Sample	DT (°C)	AC (mg/g DM)	TPC (mg GAE/g DM)	RED (EC_{50}, mg DM/ml)	AA (EC_{50}, mg DM/ml)	CHEL (EC_{50}, mg DM/ml)
FP	–	18.62 ± 0.23[h]	14.3 ± 0.27[f]	5.4 ± 0.11[a]	19.3 ± 0.22[a]	18.7 ± 0.18[a]
FD	20	17.1 ± 0.65[d]	12.6 ± 0.32[e]	6.1 ± 0.18[b]	21.7 ± 0.47[c]	20.0 ± 0.22[b]
	40	16.6 ± 0.54[df]	12.5 ± 0.41[e]	6.0 ± 0.21[b]	22.3 ± 0.39[c]	22.6 ± 0.34[c]
	60	13.9 ± 0.44[abc]	11.8 ± 0.28[d]	6.7 ± 0.23[c]	26.1 ± 0.52[ef]	25.1 ± 0.43[e]
WB	20	13.9 ± 0.45[abc]	10.8 ± 0.26[ab]	7.1 ± 0.16[d]	26.9 ± 0.61[f]	26.6 ± 0.21[g]
	40	13.5 ± 0.43[ab]	10.6 ± 0.32[ab]	7.3 ± 0.23[d]	27.2 ± 0.76[f]	26.2 ± 0.27[g]
	60	9.7 ± 0.34[g]	10.2 ± 0.36[ab]	7.5 ± 0.26[d]	29.9 ± 0.94[g]	28.4 ± 0.29[h]
MBs	20	15.4 ± 0.46[ef]	10.9 ± 0.21[bc]	6.2 ± 0.13[b]	24.2 ± 0.61[d]	24.2 ± 0.26[e]
	40	15.0 ± 0.47[ce]	11.3 ± 0.29[c]	6.4 ± 0.19[b]	25.2 ± 0.63[de]	24.7 ± 0.34[ef]
	60	12.4 ± 0.46[a]	10.5 ± 0.27[b]	6.8 ± 0.15[c]	27.4 ± 1.12[f]	25.4 ± 0.42[f]
CA	20	17.4 ± 0.56[d]	12.1 ± 0.35[e]	6.2 ± 0.08[a]	20.6 ± 0.69[b]	23.3 ± 0.38[d]
	40	17.1 ± 0.55[d]	12.5 ± 0.23[e]	6.4 ± 0.20[ab]	22.7 ± 0.73[c]	23.5 ± 0.44[d]
	60	14.7 ± 0.63[bce]	11.6 ± 0.43[d]	6.9 ± 0.21[c]	25.1 ± 0.58[d]	22.7 ± 0.36[c]

*The values are expressed as mean ± SD (n = 3); DT – drying temperature, FP – fresh pepper, FD – freeze dried (without pretreatment), WB – water blanching, MB – microwave blanching, CA – with citric acid addition, AC – L-ascorbic acid content, TPC – total phenolics content, RED – reducing power, ABTS – antiradical activity, CHEL – chelating power. The values designated by the different small letters in the columns of the table are significantly different (α = 0.05).

paprika colour quality. Moreover, higher temperature of drying can also increase the rate of carotenoids degradation (Vega-Gálvez, Lemus-Mondaca, Bilbao-Sáinz, Fito, & Andrés, 2008). Dried pepper with the addition of CA was more red and was characterized by the highest values of colour intensity in comparison to dried material without acid addition. Moreover, blanching caused a slight decrease in the redness and increase in the yellowness of pepper, whereas the method of blanching had little or no effect on colour coordinates. Wang et al. (2017b) found similar results after blanching pepper before air drying.

3.3. L-ascorbic acid changes

The L-ascorbic acid content (AC) of fresh pepper was 18.6 mg/g DM. L-ascorbic acid can be easily degraded, depending on many factors such as temperature, pH, light, and presence of enzymes and/or oxygen, Thus, many studies on food processes take vitamin C as a quality indicator of the foods (Santos & Silva, 2008). The variations in AC content during pepper drying are shown in Table 5. Freeze-drying the control samples and the samples of pepper with CA caused the lowest decrease in AC when compared to peppers before drying. The maximum AC was obtained after drying the peppers at 20 °C and 40 °C. The increase in the drying temperature from 20 °C to 40 °C had no significant effect on the AC. With an increase in drying temperature up to 60 °C, a decrease in AC was observed (from 14% for samples with CA to 18% for WB pepper). The lowest values of AC were obtained for samples blanched before freeze-drying. However, for the WB samples, the highest decrease in LA was observed. Another study confirmed that blanching the pepper significantly decreased the AC (Castro et al., 2008). A study by Wang et al. (2017a) showed that WB pepper in comparison with MB was characterized by the lowest retention of L-ascorbic acid. We also found positive correlation between the redness of the dried pepper and AC (r = 0.820, p = 0.001).

3.4. Total phenolics content and antioxidant activity

The phenolics content (TPC) of fresh pepper was 14.27 mg GAE/g DM. The variation in the TPC content during pepper drying at different temperatures is shown in Table 5. The freeze-drying caused a decrease in TPC. The maximum TPC was obtained during the drying of untreated pepper at 20 °C and 40 °C, and for samples with CA addition (average 12.4 mg GAE/g DM). With an increase in drying temperature from 40 °C to 60 °C, a decrease in TPC was observed. The lowest values of TPC was obtained for blanched peppers (from 10.2 to 11.3 mg GAE/g DM). The method of blanching had little effect on TPC. Slightly higher values of TPC were obtained for MB of pepper than for WB fruits. However, the significant differences were obtained only when the temperature of FD was 40 °C. The antioxidant activities (RED, AA, CHEL) of fresh pepper expressed as EC_{50} were 5.4, 19.3 and 18.7 mg DM/mL, respectively. The freeze drying caused a decrease in RED, AA, and CHEL. The highest EC_{50} values (the lowest antioxidant activity) were obtained for WB and dried pepper (average 7.4, 28.0 and 27.1 mg DM/mL, for RED, AA and HEL, respectively), whereas the lowest EC_{50} were found for dried pepper without pretreatments and with the addition of CA (6.4, 23.1 and 22.9 mg DM/mL, for RED, AA and HEL, respectively) (Table 5). In comparison to WB samples, the MB samples of dried pepper were characterized by higher antioxidant activity. Wang et al. (2017a) showed that MB can enhance the antioxidant activity of pepper, whereas WB had an adverse effect because of leaching of hydrophilic antioxidants into the hot water. An increase in drying temperature from 20 °C to 40 °C had no significant effect on antioxidant activity. However, when the drying temperature was 60 °C, a slight but significant decrease in antioxidant activity was observed. Many authors found that the processing temperature significantly influenced the antioxidant capacity of pepper. Rufián-Henares, Guerra-Hernández, and García-Villanova (2013) found that a higher temperature of dehydration exerts a strong effect on the degradation of antioxidant compounds. Zhou et al. (2016) showed that during hot air drying, the total phenolics content and antioxidant activity in red pepper decreased proportionate to increases in the drying temperature.

4. Conclusions

Blanching of pepper before freeze-drying significantly reduced the drying time (to approximately 30% of the original), whereas the addition of CA had no significant effect on drying kinetics. An increase in freeze-drying temperature from 20 °C to 60° approximately halved the drying time and produced paprika with a lower lightness, redness, and yellowness. The highest values of redness and yellowness of dried product were obtained for pepper with CA addition. Freeze-drying caused a decrease both in TPC and antioxidant activity. The highest decrease was observed for blanched pepper. Taking into account the drying temperature and the methods of pretreatments, the highest quality dried product (redder and with the highest TPC, AA, and L-ascorbic acid content) was obtained when CA was added to the pepper before freeze drying and when the temperature of the process was 40 °C.

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Effects of drying and grinding in production of fruit and vegetable powders: A review

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ABSTRACT

In recent years, fruits and vegetables have received considerable attention, as these materials have been reported to contain a wide array of phytochemicals, which are claimed to exert many health benefits including antioxidant activity. In some cases where bioactive compounds extraction cannot be performed on fresh products, drying appears as a necessary step enabling their later use. Drying is a widely used food preservation process in which water removal minimize many of the moisture-driven deterioration reactions impacting the bioproduct quality. Dried fruits and vegetables and their application in powder form have gained interest in the food industry. Drying and grinding conditions during powder processing greatly influence the quality attributes of biological materials. It implies not only nutritional changes but also physical, textural, sensorial and functional changes. These changes are of great importance and require to be controlled through retroengineering approaches. This paper reviews the effect of the different dry drying and grinding methods on the physicochemical and functional properties of the final products. Overviews of some of the innovative concepts as well as approaches to alleviate the above-mentioned changes are discussed.

Contents

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Nomenclature:

ADG	Alternation of Drying and Grinding
CD	Convective Drying
DD	Desiccant Drying
DIC	Détente Instantanée Contrôlée
DIS	Dewatering Impregnation Soaking in concentrated solutions
EHD	ElectroHydrodynamic Drying
FD	Freeze Drying
HAD	Hot Air Drying
HPD	Heat Pump Drying
IRD	Infrared Drying
MW	Microwave
MWD	Microwave Drying
MWAD	Microwave-assisted air drying
MWFD	Microwave-assisted freeze drying
MWVD	Microwave-assisted vacuum drying
OD	Osmotic Dehydration
OH	Ohmic
PEF	Pulsed Electric Field
RFD	Radiofrequency Drying
RWD	Refractance Window Drying
$scCO_2D$	Supercritical Carbon Dioxide Drying
SSD	Superheated Steam Drying
VD	Vacuum Drying

1. Introduction

Fruits and vegetables have proved to be essential for a balanced diet. Epidemiological and clinical investigations have actually associated diets rich in fruits and vegetables with reduced risks of cardiovascular, coronary heart, metabolic and degenerative diseases, as well as certain form of cancers (Saleem et al., 2002; Zhang et al., 2005a,b; Dai et al., 2006; Chen et al., 2006). This is believed to be mainly due to their content in fibers, vitamins, minerals and phytochemicals, such as polyphenols, flavonoids, sterols, carotenoids, chlorophylls, anthocyanins, etc., responsible in part for their strong antioxidant activity (Andersen and Jordheim, 2006; Siriamornpun et al., 2012). Indeed, these bioactive compounds are known to chelate metal ions (such as iron and copper), donate hydrogen atom and scavenge harmful free radicals associated with chronic diseases, protecting thus human tissues and cells from oxidative damages (Edge et al., 1997; Heim et al., 2002; Johnson, 2002; Balasundram et al., 2006). Fresh fruits and vegetables are highly perishable commodities (due to their high moisture content around 80%) that deteriorate over a short period of time if improperly handled (Orsat et al., 2006). Drying fruits and vegetables is a process where water removal halts the growth of spoilage microorganisms, as well as the occurrence of enzymatic or nonenzymatic browning reaction in the material matrix (Zhang et al., 2006; Argyropoulos et al., 2011; Kurozawa et al. 2012) preserving thus the structure, sensorial characteristics and nutritional value of the starting material (Aguilera, 2003). The market for dehydrated fruits and vegetables has actually known a rapid growth rate (of 3.3%) for most countries worldwide (Zhang et al., 2006). Dried fruits and vegetables are widely used by the confectionary, bakery, sweet and distilling industries in various sauce, teas, puddings, garnishments and food for infants and children. Applications particularly include fruits and vegetables powders used as intermediate products in the beverage industry, as functional food additives improving the nutritional value of foodstuff, as flavoring agent (in ice creams, yogurts, fruit bars) or also as natural colorants (Camire et al., 2007). Camire et al. (2007) have described for example a more attractive white cornmeal breakfast cereal when blueberry and cranberry fruit powders were added as colorants. Correia da Costa et al. (2009) have highlighted the usefulness of guava and cashew-apple powders in food industry as high dietary fiber ingredients fortifiers. Fruit and vegetable powders likewise serve as ingredients in instant noodles, dried soups and other food recipes (Nindo et al., 2003a,b; Argyropoulos et al., 2011; Zhang et al., 2012). Their use in perfumery and cosmetics (such as *Kaempferia galanga* powder for instance) as well as resources for nutraceutical has also been reported (Correia da Costa et al., 2009; Chan et al., 2009). The quality of a fruit/vegetable powder is highly dependent upon the drying/grinding medium and conditions, as well as the composition, physical properties, production system (conventional or organic) and cultivar-field (mechanically or hand harvested) of the raw material (Sablani, 2006; Rahman et al., 2009; Sablani et al., 2011). Quality degradations like shrinkage, puffing, crystallization, decrease in rehydration capacity and losses of taste, aroma, color and nutritional values are the main problems encountered and to be solved through dry drying and grinding processes (Devahastin and Niamnuy, 2010; Sablani, 2006; Zhang et al., 2012). This paper primarily aims at comparing the different (traditional and novel) dry drying processes. Some approaches to minimize the adverse effects of processing and enhance the quality of final products are also discussed.

2. Effect of dry drying methods on overall quality of fruit and vegetable powders

2.1. Dry drying of fruits and vegetables

Drying is one of the oldest, most common and most diverse food processing methods. It is a complex process involving simultaneous heat and mass transfer requiring precise process control (Mujumdar & Passos, 2000). Drying a moist material implies evaporation of both free and loosely bound water from inside the solid material into the atmosphere. The latent heat of vaporization may be supplied by convection, conduction and radiation or volumetrically in situ by placing the wet material in microwave or radiofrequency electromagnetic fields (Mujumdar, 2007). Drying is energy-intensive process accounting for 10–25% of the total energy used in the food manufacturing process worldwide (Strumillo and Adamiec, 1996). Fruits and vegetables are usually dried to extend shelf-life, enhance storage stability, minimize packaging requirements and reduce transport weight. Numerous processing techniques have been used for dry drying of fruits and vegetables (Ahmed, 2011). Conventionally, fruits and vegetables are sun- or hot-air-dried. Traditional solar drying is often a slow process impeded by haze, high humidity (rain), windblown debris, weather uncertainties, insect, rodents and birds infestation (Ringeisen et al., 2013), where the poor quality (in terms of color, nutritional composition and hygiene) has led to the development of alternate drying technologies (Mosha et al., 1995; Gallali et al., 2000; Ramana Murthy, 2009). Hot air-drying offers dehydrated products that can have an extended shelf-life of a year, but unfortunately with a drastically reduced quality from that of the original foodstuff. Freeze drying is a gentle dehydration technique, representing the ideal process for the production of high-value products. Vacuum drying is an important dehydration method usually used for high-value and heat-sensitive fruits and vegetables. Osmotic dehydration is an attractive method for partially drying food products, resulting in minimally processed foods. Drying in microwave (MW) field is another dehydration technique offering the opportunity to reduce the drying time and improve the quality of a dehydrated product (Maskan, 2001). Combination drying (also known as hybrid drying) is an alternative booming drying technique where the combination of processes can favor complementary process benefits. Other new drying methods (barely used at industrial level) such as desiccant drying, infrared drying and supercritical carbon dioxide drying, etc., have also been reported in the recent scientific literature and will be briefly described in this manuscript (Mujumdar, 2007).

First of all, it appears important to highlight that drying behavior of food materials largely depends both on external conditions, such as temperature (in direct relation with the glass transition temperature of food material), pressure, relative humidity, and air velocity, and on internal factors evolving in the course of drying, like food material composition, moisture content, thickness and geometry, initial structure, water diffusivity, as well as its physical (glassy or rubbery) state (Khraisheh et al., 1997; Mulet et al., 2000; Ratti, 2001; Hatamipour and Mowla, 2002; Mayor and Sereno, 2004; Gornicki and Kaleta, 2007; Yadollahinia and Jahangiri, 2009). In this paper, the effects of some dry drying methods and their combination on the most common physical (color, appearance, particle size and shape), textural, structural (density, porosity, specific volume, etc.), sensorial (aroma, taste, flavor), nutritional (vitamin, phytochemical content, etc.), and functional properties (rehydration capacity, antioxidant activity, powder flowability, etc.) of the final products are described. It is important to point out that this bibliographical study concerns the dry drying methods only: wet drying methods such as spray drying, drum drying, foam drying, etc., will not be addressed.

2.1.1. Convective drying

Conventional drying, also referred as hot-air (HAD) or convective drying (CD) is the most economical and widely adopted technique in the food industry, although requiring long drying times and high air temperatures. In air drying, the heated air (of low relative humidity) meets the surface of the wet material that transfers heat into the solid primarily by conduction. The liquid migrates then onto the material surface and is transported away by air convection. Transport of moisture within the solid food occurs by liquid or vapor diffusion, surface diffusion, hydrostatic pressure differences and combinations of these (Ahmed, 2011). HAD usually occurs in two stages, each characterized by a different drying rate. In the early stage, free water moves to the surface and is easily removed by vaporization. Then, as the drying progresses, drying becomes difficult (the liquid phase contained in solid materials becomes more viscous) and the drying rate declines (it takes more time for the internal moisture to move to the surface) (Nijhuis et al., 1998; Tsami et al., 1998; Ratti, 2001; Askari et al., 2009; Argyropoulos et al., 2011; Horszwald et al., 2013). Usually, around two thirds of the drying time may be spent removing the last one third of the moisture content, mainly corresponding to loosely-bound water molecules (Andres et al., 2004). However, it is important to point up that in the case of some green leafy vegetables such as coriander leaves, mint leaves and spinach, moisture evaporation is very rapid and completed within an hour or two (Ahmed, 2011). It seems important to mention that throughout drying, the involved diffusion transport mechanisms have a significant role, especially in the falling rate period that is controlled by mechanisms of liquid and vapor diffusion (Ahmed, 2011). This behavior indicates an internal mass transfer-type drying, with moisture diffusion as the controlling step. It is generally difficult to predict mass diffusion coefficients theoretically; therefore, experimental techniques based on sorption/desorption kinetics, moisture content distribution, or porosity have been used (Ahmed, 2011). Besides, among fitted equations, Fick's second law of diffusion equation is commonly used to describe moisture transport during drying (Ahmed, 2011).

Upon water loss, food materials undergo volumetric changes termed shrinkages and collapses. Many authors have reported a severe shrinkage (Chua et al., 2000; Krokida et al., 2000a,b; Ratti, 2001; Mayor and Sereno, 2004; Mrad et al., 2012; Russo et al., 2013) during hot-air drying up to 80% for berries and kiwi for instance according to Jankovié (1993) and Maskan (2001). Karathanos et al. (1996) have found that the severe collapse of air-dried plant materials was proportional to the moisture content being lost during the process. Furthermore, the loss of water causes tensions in cellular structure resulting in a decrease in cell size, roundness, compactness and an increase in elongation as observed when drying chestnuts (Moreira et al., 2010), carrots (Kerdpiboon et al., 2007), red peppers (Vega-Galvez et al., 2009) and apples (Mayor et al., 2005). Air-dried products are characterized by the lowest porosity and highest apparent density among the other drying methods (Lewicki, 1998; Tsami et al., 1998; Chua et al., 2000; Ratti, 2001; Krokida et al., 2000a,b; Askari et al., 2009; Witrowa-Rajchert and Rzaca, 2009; Argyropoulos et al., 2011; Lopez-Ortiz et al., 2013, Russo et al., 2013). Moreover, the color deterioration (which might be due to Maillard reactions) exhibited during drying was the most pronounced in hot-air-dried materials with a remarkable decrease in lightness and increase in yellowness values (Chen and Martynenko, 2013) as observed for air-dried asparagus (Nindo et al., 2003a,b), mushrooms (Argyropoulos et al., 2011) and apricots (Karabulut et al., 2007). A dense microstructure with a tight packing and strong connection between cells as well as a hard texture were reported for air-dried materials (Argyropoulos et al., 2011). An inability of air-dried plant tissues to imbibe sufficient water and fully rehydrate during rehydration was observed by many authors (Jayaraman et al., 1990; Ratti, 2001; Kaymak-Ertekin, 2002; Zielinska and Markowski, 2012). Krokida and Philippopoulos (2005) described the irreversible structural changes occurring during HAD and the subsequent hysteresis state upon rehydration of different dried fruits and vegetables: green pea, carrot, banana, corn, apple, potato, pepper, onion, mushroom, leek, garlic, tomato and pumpkin. From their part, Lin et al. (1998) specified the lowest ratings for aroma, texture, color and appearance in sensory evaluation of air-dried carrot slices.

Regarding the phytochemicals content, Nicoli et al. (1999) have found that carotenoid compounds, more particularly lycopene, were heat-stable, even after severe heat treatments. On the contrary, Shi et al. (1999) have demonstrated that lycopene retention is reduced in conventional air-dried tomatoes. Moreover, some authors have reported that HAD reduced the total carotenoid concentration (around 19% for both α- and β-carotene) as seen in air-dried carrot slices, paprika and sweet potatoes (Lin et al., 1998; Minguez-Mosquera et al., 2000; Yang et al., 2010). From their part, Zainol et al. (2009) have described losses up to 97% (for

Centella asiatica for example) of flavonoids content (flavonols, flavones and catechins) during HAD, the catechins fraction being the least affected by HAD. An important depletion of vitamin C varying from 20% to 60% was observed during HAD for several agricultural materials (Lin et al., 1998; Dewanto et al., 2002; Yang et al., 2010). Besides, Katsube et al. (2009) and Yang et al. (2010) have reported a decrease in phenolic content and anthocyanin concentration (from 20% to 80%) during HAD. In contrast, many authors have observed an increased phenolic concentration in several air-dried products (including sweet potato and strawberry), up to 40% in red raspberries and 54% in sweet corn for example (Dewanto et al., 2002; Yang et al., 2010; Sablani et al., 2011). As for antioxidant capacity, it was found to be enhanced in air-dried fruits and vegetables (from 29 to 33%) (Nicoli et al., 1997; Nindo et al., 2003a,b; Del Caro et al., 2004; Sablani et al., 2011) and up to 44% according to certain authors (Dewanto et al., 2002). On the contrary, a significant decrease (around 70%) in the antioxidant activity during HAD of blueberries and raspberries has been reported by others (Sablani et al., 2011). Papettl et al. (2006) concluded that HAD might enhance or deplete the antioxidant activity depending on the substrate nature. The former finding has been ascribed to polyphenols with intermediate oxidation state (which usually exhibit a higher scavenging activity than non-oxidized polyphenols) and to the formation of Maillard reaction products acting as pro- or antioxidants (Nicoli et al., 1999; Piga et al., 2003; Mrkìc et al., 2006).

It is important to highlight that the air-drying temperature is one of the most important factors determining the quality of the end product (Larrauri et al., 1997; Gupta et al., 2011; Chen and Martynenko, 2013). Indeed, although high drying temperatures (from 60 to 80 °C) result in an exponential increase in drying rates, it induces undesirable quality degradations (Karim and Hawlader, 2005; Vega-Galvez et al., 2009; Seiiedlou et al., 2010; Russo et al., 2013). Cracks, casehardening, total structure breakage, marked color deterioration, important phytochemical depletion, significant antioxidant activity reduction, as well as considerable shrinkage rates are the main damages occurring during HAD at high temperatures (Minguez-Mosquera et al., 2000; Ramos et al., 2003, 2004; Hu et al., 2006; Mrad et al., 2012; Kurozawa et al., 2012; Russo et al., 2013).

It is worth noting that shrinkage phenomenon is closely related to negative quality attributes of the dried material since shrunken products are characterized by increased hardness and biochemical reactions, reduced bulk density, poor appearance, plus a reduced ability of water retention during rehydration (Krokida and Philippopoulos, 2005; Askari et al., 2009; Argyropoulos et al., 2011; Oikonomopoulou and Krokida, 2013).

2.1.2. Vacuum drying

Vacuum drying (VD) is a process in which moist material is dried under sub-atmospheric pressures (Arevalo-Pinedo and Murr, 2005). During vacuum drying, water molecules diffuse to the surface and evaporate into the vacuum chamber. The partial vacuum in the drying chamber reduces water vapor concentration at the product surface generating thus a vapor pressure gradient (Dev and Raghavan, 2012). Heat is usually supplied by conduction to the system at a partial vacuum of about 50– 100 mbar to achieve the best product quality. Vacuum drying enables the products to be dried at low temperature (below 75 °C; usually at temperatures close to 30 °C), in the quasi absence of air and at faster drying rates (Lin et al., 1998; Markowski and Bialobrzewki, 1998; Gunasekaran, 1999; Bazyma et al., 2005). It should be mentioned that VD appears as the method providing the largest driving force for mass transfer according to many authors (Drouzas et al., 1999; Jaturonglumlert and Kiatsiriroat, 2010). VD is particularly suitable for the dehydration of heat-sensitive and easily oxidizable products (Jaya and Das, 2003; Dev and Raghavan, 2012; Quintero-Ruiz et al., 2013). In the food industry, vacuum drying is generally carried out in conjunction with some other techniques, like microwave-vacuum drying or vacuum freeze-drying for example. Applying VD engenders products with higher porosity, lower apparent density, as well as lower shrinkage rates than hot air-dried materials (Jaya and Das, 2003; Arévalo-Pinedo and Murr, 2005, 2007; Wu et al., 2007; Sahari et al., 2008; Oikonomopoulou and Krokida, 2013). Moreover, vacuum-dried materials displayed less color deterioration and larger pores than conventionally dried foodstuffs (Jaya and Das, 2003; Orikasa et al., 2008; Quintero-Ruiz et al., 2013). Furthermore, this technique seems to be an appropriate method for protecting ascorbic acid (high retention level of 44%) (Inyang and Ike, 1998; Methakhup et al., 2005; Orikasa et al., 2013), phenolic compounds (retention up to 25.1%) (Quintero-Ruiz et al., 2013), anthocyanins (Witrova and Rzaca, 2009), lycopene and more particularly transisomers (retention up to 94%) (Shi et al., 1999). Also, it allows preserving the substantial antioxidant activity of these compounds (Quintero-Ruiz et al., 2013). Oriskasa et al. (2013)) have even described a five time higher antioxidant activity in vacuum dried kiwifruits slices. Finally, this technique remains, however, too costly for consideration at large production scales (Motevali et al., 2011).

2.1.3. Freeze-drying

Freeze-drying (FD) also known as lyophilisation is a gentle dehydration technique representing the ideal process for the production of high-value dried products. This technique is well-known for its ability to maintain the product quality (color, shape, aroma and nutritional value) greater than any other drying method, owing to both its low processing temperature (from − 2 to − 10 °C) and the virtual absence of air oxygen during processing, which minimize degradation reactions (Strumnillo and Adamiec, 1996; Litvin et al., 1998). Other prominent factors include the structural rigidity exhibited by the frozen substance at the surface, as well as the limited mobility of frozen water preventing thus collapses and shrinkages of the solid matrix. The FD process mainly consists in two steps where the product is first frozen (−20 °C) then a controlled amount of heat under vacuum (vacuum freeze-drying) or at atmospheric pressure (atmospheric freeze-drying) is applied to promote sublimation (Oikonomopoulou and Krokida, 2013). During FD, the removal of internal moisture passes through two stages: an initial one where ice crystals sublimation occurs and a secondary stage where desorption of the unfrozen remaining water occurs. Freeze-dried materials are characterized by the lowest values of apparent density and the highest porosity (Ratti, 2001; Krokida et al., 2000a,b; Askari et al., 2009; Witrowa-Rajchert and Rzaca, 2009; Argyropoulos et al., 2001; Russo et al., 2013; Oikonomopoulou and Krokida, 2013). Minimal shrinkage (5–15% for berries for example) and negligible collapse (less than 10%) have been observed for most types of foodstuffs during freeze-drying (Jankovié, 1993; Ratti, 1994; Karathanos et al., 1993, 1996; Chua et al., 2000; Ratti, 2001; Mayor and Sereno, 2004; Koc et al., 2008; Askari et al., 2009; Witrowa-Rajchert and Rzaca, 2009; Argyropoulos et al., 2011; Horzswald et al., 2013; Russo et al., 2013). It is important to mention, however, that the fraction of collapsed food sample during FD can increase with the plate heating temperature as observed for celery and potatoes (around 25%) for example and can reach values as high as 45% as observed in freeze-dried apples (Karathanos et al., 1996; Ratti, 2001). Indeed, many authors have related the more intense shrinkage exhibited when FD takes place at higher temperatures. Others have linked the highest porosity of FD materials to the lowest FD temperatures (Krokida et al., 1998; Oikonomopoulou and Krokida, 2011; Rahman and Sablani, 2003). On the other hand, freeze-dried materials rehydrate more completely than other samples and display the fastest rehydration rates as well as the highest rehydration ratio (Giri and Prasad, 2007; Antal et al., 2011; Argyropoulos et al., 2011). Ratti (2001) has precised a rehydration ratio up to 4–6 times higher for freeze-dried than air-dried products, making freeze-dried products excellent for ready-to-eat meals or soups. This improved water potential reconstitution has been mainly explained by the porous structure observed in freeze-dried materials (Ratti, 2001; Argyropoulos et al., 2011; Oikonomopoulou and Krokida, 2013). Moreover, freeze-dried products yielded the minimal color deterioration and the highest lightness, as well as the lowest yellowness values, highlighting once again the relevance of this process to preserve nutraceutical components (Nindo et al., 2003a,b; Argyropoulos et al., 2011).

The loss of bioactive compounds, such as total flavonoids, flavonols, flavones, catechins, as well as phenolics was further found to be negligible in freeze-dried samples (Asami et al., 2003; Zainol et al., 2009). Some authors have even reported an increase in total phenolic and anthocyanin concentrations by 17–52% and 7–26% respectively, the level of increase primary depending on the material type (blueberries, raspberries, *Alpinia Zerumbet* (ginger), or Elatior leaves), production system and cultivar-field pair (Chan et al., 2009; Sablani et al., 2011). An almost complete retention of carotene, 96% as specified by Abonyi et al. (2002), was observed during FD of carrots, starfruit, papaya, muskmelon, and strawberries (Abonyi et al., 2002; Regier et al., 2005; Shofian et al., 2011). Yang et al. (2010) have further described similar β-carotene content for freeze-dried sweet potatoes and fresh samples. It worth noting, however, that some authors have reported a decrease in β-carotene concentration by 26% and 43% as seen in freeze-dried mango and watermelon respectively (Shofian et al., 2011). In addition, many authors have reported a maximal (up to 94% in certain cases, and in all cases higher than 60%) ascorbic acid retention (Santos and Silva, 2008) as observed in freeze-dried guava (Nogueira et al., 1978), tomatoes (Chang et al., 2006), sweet peppers (Martínez et al., 2005), seaweed (Chan et al., 1997), strawberries (Abonyi et al., 2002), carrots (Lin et al., 1998), asparagus (Nindo et al., 2003a,b), mango, starfruit, watermelon and finally muskmelon (Shofian et al., 2011). Besides, the antioxidant activity was found to be increased by at least 13% (till 82%) in several freeze-dried products (Sablani et al., 2011; Shofian et al., 2011).

Finally, it is important to highlight that contrariwise, this technique appears as a very lengthy and expensive preservation method because of the low drying rates spawned by refrigeration and vacuum systems that increases energy costs (Liapis et al., 1996; Ratti, 2001). Mafart (1991) and Ratti (2001) have quantified FD costs to be 4–8 times higher than that of HAD. As such, the use of FD on the industrial scale seems to be restricted to high-value products and so far to instant coffee, as well as edible and medicinal species (Lin et al., 1998; Yang et al., 2010).

2.1.4. Microwave drying

Microwave (MW) drying (MWD) is an alternative drying method gaining popularity in recent years for a wide variety of industrial food products (Krokida et al., 2000a,b). It can be regarded as a rapid dehydration process significantly reducing the drying time, up to 89% of the HAD time according to certain authors (Maskan, 2001; Therdthai and Zhou, 2009). A MW drying process consists in three drying periods: (1) a heating-up period in which MW energy is converted into thermal energy within the moist materials and the product temperature increases with time, (2) a rapid drying period during which thermal energy is used for moisture vaporization and transfer and (3) a reduced drying rate period during which the local moisture is reduced to a point that the energy needed for moisture vaporization is lower than the thermal energy induced by MW (Maskan, 2001; Zhang et al., 2006; Ozyurt et al., 2011). MW drying can be assigned as a "volumetric heating process", MW electromagnetic energy being directly absorbed by water-containing materials and converted into heat by molecular agitation (Khraisheh et al., 1997; Piyasena et al., 2003). It is worthy to note that MWD has been demonstrated to have moderately low energy consumption (Tulasidas et al., 1997; Sagar and Kumar, 2010; Motevali et al., 2011). MWD (usually at frequencies of 915 and 2450 MHz) has been used in drying of herbs (Özbek and Dadali, 2007), potatoes (Bouraoui et al., 1994), raisins (Kostaropoulos and Saravacos, 1995), apples and mushrooms (Feng and Tang, 1998), carrots (Jia et al., 2003), kiwifruits (Maskan, 2001), asparagus (Nindo et al., 2003a,b), pears (Kiranoudis et al., 1997), American ginseng roots and bananas (Maskan, 2000). In general, MW application has been reported to improve the overall product quality with great aroma, color and nutrients retention, relatively fast rehydration rates, as well as considerable savings in energy (Gunasekaran, 1999; Maskan, 2000; Torringa et al., 2001; Orsat et al., 2007; Ghanem et al., 2012). From its part, Maskan (2001) has described a high shrinkage rate (around 85%) and a low rehydration capacity in MW-dried kiwifruit slices. Furthermore, Park (1987) has quantified the loss of carotenoids (around 63%) during MWD of carrots. Many authors have actually highlighted several drawbacks of single MW drying: namely, uneven heating (occurrence of hot and cold spots in the foodstuffs during heating subsequent to nonuniform electrical field), textural damages (especially, arcing at power above 500 W in small-scale drying cavities, and puffing), as well as subsequent scorching and development of off-flavors (Nijhuis et al., 1998; Erle and Schubert, 2001; Zhang et al., 2006). Raghavan and Venkatachalapathy (1999) have further described the "burn" of MW-dried strawberries despite the low applied power of 600 W. Giese (1992) and Zhang et al. (2006) revealed that MWD seems to be the most effective for products of moisture content below 20%.

The combination of MWD with other drying methods permits to overcome some limitations of single MWD. In combined drying methods, MW is particularly suitable for the drying of heat-sensitive materials and offers the opportunity to significantly shorten drying time and enhance product quality as compared with hot-air- or single MW-dried products (Schiffmann, 2001; Zhang et al., 2006; Heindl and Müller, 2007). MW-assisted drying techniques can be divided into: MW-assisted air drying (MWAD), MW-assisted vacuum drying (MWVD) and MW-assisted freeze drying (MWFD).

2.1.4.1. Microwave-assisted air drying. Microwave-assisted air drying (MWAD) is principally used in several industrial food processing applications instead of HAD in order to shorten drying time, improve food quality and prevent shrinkage of tissue structures (Feng et al., 2001; Schiffmann, 2001). There are three methods in which MW energy might be combined with HAD (Andrés et al., 2004) (1) by applying the MW energy at the beginning of the dehydration process: the interior of the product is in this case quickly heated to the evaporation temperature and water from the surface is removed; (2) by applying MW energy when the drying rate begins to fall: in this case, the material surface is already dry and moisture concentrated in the food bulk; when applying MW at this stage, the generation of internal heat and the subsequent increase in vapor pressure force the moisture to migrate to the surface and is hence removed by the ambient environment; (3) by applying MW in the falling rate period (i.e. at low moisture content) to finish drying. Applying MW drying in the last stage of the dehydration process is particularly efficient in removing bound water from the product (Zhang et al., 2006). Maskan (2001) has recommended, essentially for economic reasons, the application of

MW energy at this stage where conventional HAD usually takes too long. He further described an increased drying rate and a substantial shortening of HAD time by about 64% when applying MWD at a final drying stage. He also reported high rehydration ratios and limited shrinkage (of about 40%) for "MW finished" dried product as seen in kiwifruits and bananas slices for instance.

Some authors have carried out studies on combined convection HAD and MW-heating using potatoes, carrots, mushrooms, apples and strawberries as model materials (Funebo and Ohlsson, 1998; Funebo et al., 2002; Jia et al., 2003; Andrés et al., 2004). They found out enhanced moisture diffusivity, improved rehydration capacity and marked flavor retention (Riva et al., 1991; Askari et al., 2009). Witrowa-Rajcher et al. (2009) have stated that the use of MWAD was the most advantageous drying method for deep purple carrots variety during which a limited reduction (of approximately 20%) of anthocyanins, polyphenols and antioxidant capacity was observed (versus 50% for hot-air dried deep purple roots).

2.1.4.2. MW-assisted vacuum drying. Microwave assisted vacuum drying (MWVD) is one of the recently emerging food processing methods where vacuum drying is introduced to replace conventional HAD (Hu et al., 2006). Applying MW energy under vacuum seems especially suitable for heat-sensitive products (such as fruits of high sugar content and high-value vegetables) as far as improved energy efficiency and product quality are concerned. MWVD technique has been successfully used for drying grapes (Clary et al., 2005), cranberries (Yongsawatdigul and Gunasekaran, 1996), bananas (Mousa and Farid, 2002), tomatoes (Durance and Wang, 2002), carrots (Cui et al., 2005; Regier et al., 2005), garlic (Cui et al., 2005), potatoes (Bondaruk et al., 2007), kiwifruits, apples, pears (Kiranoudis et al., 1997), mushrooms (Giri and Prasad, 2007; Argyropoulos et al., 2011) and mint leaves (Therdthai and Zhou, 2009). Several authors have reported that MWVD technique considerably shorten drying times by 70–90% and remains faster than HAD and FD techniques (Lin et al., 1998; Regier et al., 2005; Giri and Prasad, 2007). MWVD has yielded products with lower apparent density, higher porosity as well as lower shrinkage than hot-air-dried samples (Lin et al., 1998; Therdthai and Zhou, 2009; Argyropoulos et al., 2011). Moreover, a low-density, porous and open structure with a soft texture was reported for MW vacuum-dried materials (Yongsawatdigul and Gunasekaran, 1996; Lin et al., 1998; Bondaruck et al., 2007; Giri and Prasad, 2007; Therdtai and Zhou, 2009; Argyropoulos et al., 2011). High rehydration potential (high rehydration ratios and rates), as well as minimal extent of color changes (generally characterized by a high degree of brightness and a good yellowness) were observed for MW vacuum-dried materials (Lin et al., 1998; Cui et al., 2005; Hu et al., 2006; Argyropoulos et al., 2011). Great retention levels of vitamin C (up to 84.1%), chlorophyll (up to 32.6%), α-caroten, and vitamins A, B1, B2 and B3 were described for MW vacuum-dried materials (Yanyang et al., 2004). Finally, these products were rated of equal or better quality than freeze-dried samples by a sensory panel for texture, color, flavor and overall preference in both dried and rehydrated states (Lin et al., 1998; Zhang et al., 2006).

2.1.4.3. Microwave-assisted freeze drying. Microwave assisted freeze drying (MWFD) appears as a promising technique to accelerate the drying process, improve the overall product quality and reduce the drying costs in comparison with conventional FD (Sochanski et al., 1990; Cohen and Yang, 1995; Wu et al., 2004; Duan et al., 2008). It is a potential energy-saving process (up to 54% in comparison with conventional FD), according to Huang et al. (2009), particularly convenient for products of intermediate value (normal fruits and vegetables). It might be performed in two distinct ways: (1) FD concurrently assisted with microwave application, in this case, MW field is used as the heat source supplying the needed heat of sublimation, and (2) FD assisted with MW in two consecutive separate stages where MWD (usually under vacuum) is used to finish drying (Duan et al., 2010). The very few experimental studies using MWFD have shown a 50–75% reduction in drying time, as well as an enhanced retention of volatiles compounds in addition to a better rehydration capacity (Cohen and Yang 1995; Zhang et al., 2006). MWFD is still very difficult to use in industrial applications mainly due to plasma discharge problems occurring when the electric field intensity in the vacuum chambers exceeds a threshold value. In this case, the ionization of residual gases present in the vacuum chamber leads to the appearance of a purple lightning, liable to burn the product surface (thus seriously damaging the final product), besides the considerable energy loss (Zhang et al., 2006; Duan et al., 2008).

2.1.5. Osmotic dehydration

Osmotic dehydration (OD), sometimes referred as "dewatering impregnation soaking in concentrated solutions" (DIS), is a complex dynamic mass transfer process where fruits and vegetables (whole or in pieces) are immersed for a given period of time in an osmoactive hypertonic solution to reduce the moisture content. Osmoactive solutions might be concentrated solutions of sugars (sucrose, glucose, fructose or maltodextrin), salts (e.g., sodium chloride), combinations thereof or alcohols (glycerol or sorbitol) (Torreggiani and Bertolo, 2001; Shi and Le Maguer, 2002; Pan et al., 2003; Chiralt and Talens, 2005; Mercali et al., 2011). OD is characterized by flux exchange of water and solutes permitting fruits/vegetables to lose water (mainly by diffusion) and gain solids, depending on the process conditions (Khin et al., 2005; Ramallo and Mascheroni, 2005; Shi et al., 2008; Shi et al., 2009). OD can be described as a partial dehydration process where water content reduction is in the order of 30% and can reach a maximum of 50% depending upon several factors such as concentration, temperature, osmotic medium, etc. (Moreira et al., 2010; Ahmed, 2011). Consequently, after the initial osmotic step, a conventional drying method (such as HAD, FD or MWD) is usually necessary to produce shelf-stable dried-fruits and vegetables (Nieto et al., 1998; Maestrelli et al., 2001). The use of OD as a pre-treatment ensures improvements in quality aspect in terms of color, flavor, nutrient retention as well as energy efficiency (Lemus-Mondaca et al., 2009). Many authors have actually related the reduced drying time, improved structure, rehydration capacity, porosity, maintained bulk volume (up to 60% for apples for instance) and the limited shrinkage in osmotically pretreated products in comparison to those simply dried by conventional processes (Simal et al., 1997; Erle and Shubert, 2001; Torringa et al., 2001; Funebo et al., 2002; Piotrowski et al., 2004; Moreira et al., 2010; Botha et al., 2012). From their part, Prothon et al. (2001) and Contreras et al. (2005) have described the good appearance (characterized by a limited brownish color) and the soft texture of osmotically dehydrated foodstuffs. Some authors have further described the great retention of some bioactive compounds such as vitamin C and lycopene in osmotically treated fruits, strawberries, apples and tomatoes for example (Erle, 2005; Shi et al., 1999); whereas others have reported the significant decrease in vitamin C (in the order of 78%, for osmotically treated seabuckthorn for example), carotenoid and phenolic contents (Pan et al., 2003; Araya-Farias et al., 2014). Finally, the opposite flux of water and solutes in osmotic dehydration remains the main disadvantage of the process, especially when greater similarity to the fresh fruit or vegetable is desired.

2.1.6. Innovative dry drying concepts

The innovative dry drying concepts can be described as new methods deriving from modifications of the usual drying

Table 1
Innovative concepts of drying.

Drying technique	Principle	Advantages over Conventional drying	Typical products	Physical, functional and nutritional quality of dried products	Main processing parameters	Limits	Trends and prospects
Infrared drying (IRD)	- Infrared radiation (wavelengths from 0.78 to 1000 μm) transfers thermal energy in the form of electromagnetic waves [1] - Heat is produced at the food material surface and transferred to its interior by conduction [1,2,16]	- Increased energy efficiency [1] - Uniform temperature in the product while drying [1, 3] - Up to 50% reduction in drying time [1, 2, 16] - Simple equipment requirements [2] - Low cost [2] - Environmentally-friendly[1] - Better quality of dried products [1]	- Onion [4,5,6] - Carrot [7,8,9] - Apple [10,11] - Sweet potato [12–13] - Herbal sources, pepper [2, 3]	- High sample porosity [14] - Intact microstructure [15] - Minimal color changes [15] - Firm texture [17] - Great retention of phenol compounds [18] - Minimal loss of chlorophylls [19],vitamin C [11] and β-carotene [19, 20]	Increasing surface temperature, air temperature, or IR power permits faster drying rates[1, 22, 24]	- Swelling and fracturing of the material during long exposures to infrared radiation [21, 25] - Relatively low rehydration rates [21,25] - Restrained application to instant food formulation [21]	- Combination of IR and HAD or FD [21] - Use of catalytic IRD technique as a variant where direct conversion of natural gas to radiant energy by an intermediate catalytic reaction is more energy-efficient than the typical IR emitters [21]
Desiccant drying (DD)	- Drying at low/moderate temperatures, usually below 40 °C (i.e. at temperatures 5 or 10 °C above the ambient temperature) [16]. - Drying using dehumidified air [16] - The material to dry has to be put in contact with a moisture-absorbent material (silica gel, zeolites, borax or bentonites) for the desired water adsorption-desorption to take place [25, 26] - Saturated adsorbents are regenerated by passing through a stream of hot air absorbing the moisture[3,27]	- Easy-to-design systems [3] - Excellent product quality in terms of color, texture, and nutrient retention [3] - Combining the DD with other drying systems results in significant reduction of energy consumption, drying time, and increased energy efficiency [3, 27, 28]	- Mushrooms [25, 29] - Cabbage, eggplant, spinach [25] - Flowers [3]	- ntegral product structure [3] - Limited collapse [24] - Great rehydration abilities of dried products [3, 27] - Excellent color preservation [25, 27] - Important retention of vitamin C and aroma [25]	- Temperature and airflow control result in faster drying rates in some systems [25] - Initial moisture content of the sorbent has a strong effect on the change of product moisture (drying is improved at low initial moisture content of the sorbent) [30]	- In the desiccant-based systems proposed to date, only temperature control is allowed, resulting in low drying velocity and a heterogeneity of drying rate according to the location in the drying room, making it difficult to obtain uniform product quality [25]	- Use of adsorbents in fluidized bed and solar drying systems [29, 30] - New method of DD using pulsating airflow [25, 31]
Refractance Window drying (RWD)	- In this drying method, thermal energy from hot water is transferred to the wet material deposited as thin film on a plastic conveyor belt - Heat transfer is mainly achieved by conduction and radiation from water to the sample, and by convection from the heated sample to the surrounding air [31,32]	- Lower installation and operation costs [33] - Shorter drying times [34,35] - Reduction from 4 to 6 decades of microbial load [36] - Better quality of dried products [21]	- Carrot [35] - Asparagus [37] - Strawberry (puree) [36] - Herbs (aloe vera) [23]	Excellent retention of α- and β-carotene, as well as vitamin C (about 94%) [35, 37]	Increasing water temperature enhances the drying rate [36]	- Low capacity of the system [21] - Inconvenient to handle powders with high sugar contents [21]	Addition of glycerols in order to increase the working temperature of water was suggested but not tested [36]
Supercritical carbon dioxide drying (scCO$_2$D)	Moisture removal under supercritical pressure and temperature, typically 8–10 MPa	- Low cost of materials [39] - Drying at moderate temperatures with	Carrot [40]	- Preservation of the original structure, shape, as well as the cross-	Increasing the temperature increases the drying rate [40]	Samples are susceptible to undergo post-drying shrinkage [40]	Using modified scCO$_2$ drying with ethanol has a potential for

Table 1 (*continued*)

Drying technique	Principle	Advantages over Conventional drying	Typical products	Physical, functional and nutritional quality of dried products	Main processing parameters	Limits	Trends and prospects
	and 20–50 °C, using a supercritical fluid as dispersing agent and drying medium [38]	no toxic residues [39]		sectional geometry of the product - Limited shrinkage - Reversible color loss upon rehydration - Soft texture - Fast rehydration rates [40]			obtaining high-quality products [40]
Superheated steam drying (SSD)	Superheated steam (at a temperature higher than the vapor saturation temperature at a definite pressure) is provided to accomplish drying [16]	- Higher energy and drying efficiencies [41,42] - No oxidative or combustion reactions, leading to better quality product [42,43]	- Potato and basil [44] - Pepper seed [45] - Indian gooseberry[46]	- No casehardening [41] - Good textural properties [6] - Important retention of original volatiles compounds [44] - Great scent and color [46,47] - Fair loss of β-carotene (around 20–25%) [48] - Great retention of ascorbic acid and phenolics [46, 47,49]	Increases in temperature and steam velocity at constant operating pressure increases the drying rate [50,51]	- Generally employed temperatures unsuitable for heat-sensitive materials [6, 21] - SSD systems are quite complex [16, 21] - Deteriorative effects of condensation and glass transition [6] - Limited applications [52, 53]	IR radiation can be used in combination with SSD [52]
Heat pump drying (HPD)	- HPD is based on the thermodynamic principle of vapor compression cycle [21]	- Minimal energy cost [54] - Considerable energy saving (circa 40%) [54,55] - Significant reduction of drying time [56,57,58]	- Tomato [55] - Green sweet pepper [56] - Saffron [57] - Apple, guava, potato [59] - Mint leaves [60] - Ginger [61]	- High rehydration ratios [56,59] - Important sensory scores [56] - Appreciable product appearance and structure [59] - Great retention of chlorophyll content [56] - Minor loss of ascorbic acid [56,62]	Better performance under inert gas atmosphere (under carbon dioxide or nitrogen gas atmosphere)[59]	Certain authors have reported that HPD took 1 to 1.5 times longer than HAD [16]	Development of hybrid drying (MW, IR, solar, or RF) along with HPD [24]
Radiofrequency drying (RFD)	- Volumetric heating is generated by the friction of excited molecules in the material due to the absorbed RF radiation, usually in the frequency range from 300 kHz to 300 MHz [41]	- More uniform heating [63,64] - Higher drying rates [53]	Macadamia nut [65]	Not documented	RFD is affected by the frequency, the square of the applied voltage, product dimensions, and the dielectric loss factor of the material [16]	- Application of RFD is not common - Highly capital-intensive and energy-consuming [63,65]	- This technique should be used as a supplementary drying source rather than the major one [24]
Controlled sudden decompression to vacuum (known as Détente Instantanée contrôlée) (DIC)	- This process is based on a thermo mechanical effect induced by an abrupt transition from high steam pressure to vacuum (pressure change greater than 5 bar s^{-1}) leading	- Less compact and more porous structure of dried products [67] - Reduction of energy consumption and drying time [68,69]	- Potato - Carrot - Onion [68]	- Good rehydration properties of end products [17] - Good global appreciation of the sensorial content [69]	- The use of an atmospheric air injection after decompression to a vacuum permits to avoid shrinking [68] - Increases in stepwise in saturated steam	Shriveled surface and shrinkage occurring in heat-sensitive products without modifications of the treatment cycle [68]	- Usually applied as a texturing operation for fruits and vegetables [69] - To be used as following: partial HAD-DIC- and a final HAD stage [69]

(*continued on next page*)

Table 1 (*continued*)

Drying technique	Principle	Advantages over Conventional drying	Typical products	Physical, functional and nutritional quality of dried products	Main processing parameters	Limits	Trends and prospects
	to products expansion [66]				pressure improve the color [68]		
Ultrasonic drying (UD)	UD is a process where: - ultrasonic waves produce rapid series of alternative contractions and expansions of the material to be dried, inducing cell cavitation and ensuring thus moisture removal by a mechanism of “sponge effect” - Oscillating velocities and microstreaming at the interfaces affect the shape and the thickness of the diffusion boundary layer and thus increase the convective mass transfer in the sample [70].	Effective in improving both mass transfer rates (resulting in reduced processing time, higher throughput, and lower energy consumption) and product quality (namely, phytochemical retention) [71]	- Mushroom [72] - Carrot[73], cauliflower [71] - Tropical fruits [74] - Lemon [73] and orange peel [75] - Strawberries [76] - Potatoes [77]	- Highly porous material [75] - Great rehydration properties [72] - Depending upon processing conditions: - Minimal effect or increases in ascorbic acid content [78] - Slight increases in lycopene [78] - Increases in total phenolic, flavonoid, and flavonol content [78]	- Improvement of effective moisture diffusivity and drying rates proportional to applied acoustic intensity [70,79] - Ultrasounds involve gas/solid [73], liquid/solid [72] or direct contact applications [80]	Mechanical stresses produced by high-intensity ultrasounds might induce great cellular damages, negatively affecting total polyphenol content and antioxidant activity [74]	- Beneficial use of ultrasounds in food dehydration prior to OD, [81] convective [73], freeze[82] and fluidized bed drying [71] - Ultrasound-assisted drying mostly remains a laboratory-scale technique [16]
Electric field technologies							
Ohmic heating (OH, also known as Electric resistance or Joule heating)	- OH is a process where an alternating current passes through the material generating heat and temperature rise [83] - Seen as an internal thermal generation technology (heat is internally generated by Joule effect) [84]	- Rapid and uniform temperature distribution [85] - Energy savings of 82– 97% while reducing the heating time by 90% compared to conventional heating [86]	- Potato [83,87]	- Minimal structural and nutritional changes [88] - Attractive appearance [83] - Interesting textural properties (especially, firmness) [83,88]	- Increasing the electric field strength, as well as decreasing the frequency of alternating current, increase the drying rates [83,89] - A good contact between material surface and electrodes is required [90]	Comparable electrical resistances in a two-component system are requested to ensure equal-rate heating [90]	- Commonly used as a pretreatment method for additional improvements of drying rates [16] - An effective and alternative method for blanching vegetables[88]
High-intensity pulsed electric field (PEF)	This technology is based on the application of an electric field in the form of short or high voltage pulses to a food product, as it flows between two electrodes for a short time (usually in the microsecond scale, from 10^{-4} to 10^{-2} s) [91]	Effective in increasing drying rates (shorter processing times) and production efficiency [83,91]	- Carrot [92] - Okra [93] - Potato [94,95] - Red beetroots [96]	- Increases in phenolic compounds, anthocyanins and gallic acid [91] - Confined firmness [94]	- PEF process may either be static or continuous [97] - Moderate electric field strengths (300 −400 V cm^{-1}) for effective drying rates and product quality [98]	Marked temperature increases and subsequent detrimental thermally-induced alterations in prolonged PEF treatment [94]	- This process appears mostly suitable for liquid foods [88] - PEF can be successfully combined with OD to provide improvements in mass transfer [99]
Electrohydrodynamic (EHD)	Moisture removal is ensured through high voltage application between a pointed and a grounded electrode, generating ionic wind by corona discharge [100,101]	High drying rates and low energy consumption[100]	- Carrot [100] - Mushroom [102]	- Great rehydration capacity [103] - Good carotene retention [102] - Overall improvement of the quality of the final product [102, 103]	Enhancement of drying rates, porosity and rehydration ratios of samples at increased applied voltage [102]	Decreases in effectiveness as the drying process progresses [102]	Combination of convective and EHD drying leads to an improved drying method [16]

[1] Krishnamurthy et al., 2008; [2] Nasiroglu and Kocabiyik, 2009; [3] Chua and Chou, 2003; [4] Mongpraneet et al., 2002; [5] Jain and Pathare, 2004; [6] Sharma et al., 2005; [7] Baysal et al., 2003; [8] Hebbar et al., 2004; [9] Pan et al., 2005; [10] Nowak and Lewicki, 2005; [11] Togrul 2005; [12] Sawai et al., 2004; [13] Lin et al., 2005; [14] Léonard et al., 2008; [15] Tan et al., 2001; [16] Özgül Evranuz, 2011; [17]Shi et al., 2008; [18] Boudhrioua et al., 2009; [19] Sakai and Hanzawa, 1994; [20] Namiki et al., 1996; [21] Moses et al., 2014; [; [22] Fu and Lien, 1998; [23] Ertekin and Heybeli, 2014; [24] Chou and Chua, 2001; [25] Nagaya et al., 2006; [26] Kudra and Mujumdar 2009; [27] Tatemoto et al., 2007; [28] Rahman and

Mujumdar, 2008; [29] Seyhan and Evranuz, 2000; [30] Witinantakit et al., 2009; [31] Dias et al., 2004; [32] Hodali and Bougard, 2001; [33] Dai et al., 2002; [34] Nindo and Tang, 2007; [35] Abonyi et al., 2002; [36] Nindo et al., 2003(a); [37] Nindo et al., 2003(b); [38] Sellers et al., 2001; [39] Walters et al., 2014; [40] Brown et al., 2008; [41] Tang and Cenkowski, 2000; [42] Devahastin and Suvarnakuta, 2008; [43] Devahastin and Mujumdar, 2014; [44] Barbieri et al., 2004; [45] Kozanoglu et al., 2012; [46] Methakhup et al., 2005; [47] Shibata and Mujumdar, 1994; [48] Suvarnakuta et al., 2005; [49] Cenkowski et al., 2012; [50] Iyota et al., 2001; [51] Pronyk et al., 2004; [52] Nimmol et al., 2007; [53] Mujumdar, 2007; [54] Orsat and Raghavan, 2009; [55] Queiroz et al., 2004; [56] Pal et al., 2008, [57] Mortezapour et al., 2012; [58] Lee and Kim, 2009; [59] Hawlader et al., 2006a; [60] Colak et al., 2008; [61] Hawlader et al., 2006b, [62] Ho et al., 2002; [63] Piyasena et al., 2003; [64] Marra et al., 2009; [65] Wang et al., 2014.; [66] Louka and Allaf, 2002; [67] Iguedjtal et al., 2008; [68] Louka and Allaf, 2004; [69] Albitar et al., 2011; [70] De la Fuente-Blanco et al., 2006; [71] García-Pérez et al., 2006; [72] Jambrak et al., 2007; [73] García-Pérez et al., 2009; [74] Do Nascimento et al., 2016; [75] Ortuno et al., 2010; [76] Garcia-Noguera et al., 2010; [77] Schössler et al., 2012; [78] Rawson et al., 2011; [79] Cárcel et al., 2007; [80] Gallego-Juárez et al., 2007; [81] Deng and Zhao, 2008; [82] Xu et al., 2009; [83] Lebovka et al., 2006; [84] Knirsch et al., 2010; [85] Nguyen et al., 2013; [86] Castro et al., 2004; [87] Zhong and Lima, 2003; [88] Sakr and Liu, 2014; [89] Lima and Sastry, 1999; [90] Fellows, 2000; [91] Yang et al., 2016; [92] Gachovska et al., 2008; [93] Adedeji et al., 2008; [94] Lebovka et al., 2007; [95] Arevalo et al., 2004; [96] Shynkaryk et al., 2008; [97] Wan et al., 2009; [98] Toepf et al., 2006; [99] Amami et al., 2007; [100] Alemrajabi et al., 2012; [101] Bai et al., 2012; [102] Ding et al., 2015; [103] Dinani and Havet, 2015.

conditions of conventional drying processes or adaptations of some scientific developments that find use in other areas than drying (ultrasound, MW, electric field). These methods contribute to the dehydration process by decreasing the drying time, increasing the energy efficiency or improving the quality. These methods are commodity-specific flexible but not necessarily cost-effective. A general description of some of these novel concepts and their applications with an emphasis on vegetables and fruits is given in Table 1.

It is important to mention that most of the various innovative drying methods proposed in this review were tested at a laboratory scale. However, taking the concept and making it a real process demands a close cooperation between academia and industry. It is believed that higher quality expectations of consumers would accelerate the industrialization of novel drying concepts in the near future. From their part, the Fruitis-Agritech company (France) ran by Elie Baudelaire, co-author of this work, have recently carried out a confidential central study where some of these novel concepts, namely desiccant drying and "Détente Instantanée contrôlée", were compared, at an industrial level (using different fruit and vegetable materials), to both FD and VD processes in terms of costs, energy consumption, implementation facilities, as well as final product quality. Whereas DD yielded the best powder quality in terms of sensorial characteristics, DIC appeared to be the most cost-effective and hence compromising method with considerable sensorial properties and implementation facilities. Table 2, along with Fig. 1 (a, b) illustrate the comparison of these conceivable innovative dry drying processes at industrial scale.

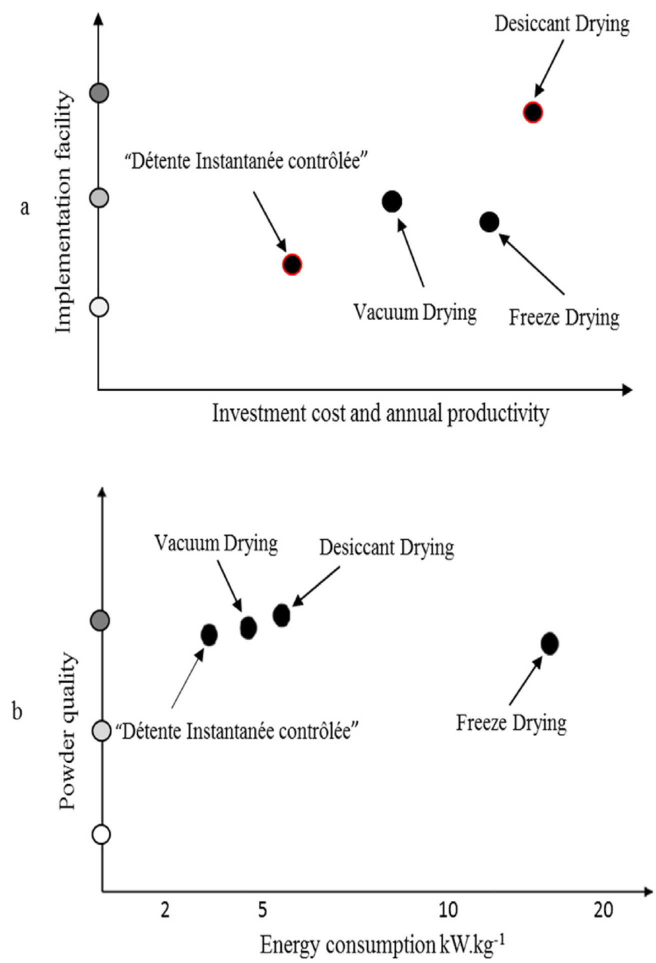


Fig. 1. Industrial comparison of Fruitis-Agritech of desiccant drying, vacuum drying, "détente instantanée contrôlée" and freeze drying.

2.1.7. Retroengineering approach of some processing parameters affecting the drying medium for improving the quality of dried fruits and vegetables

Consumers demand has increased for processed products that keep their original characteristics. This requires the development of operations that minimize the adverse effects of processing, maintain the quality of the final product, while limiting the running costs.

In HAD, air temperature, relative humidity and velocity are the main parameters that influence the final product quality. Low temperatures generally have a positive influence on the quality of biological materials requiring nevertheless long processing times, which in turn have detrimental effects on product quality and induce high costs. HAD at temperatures of 55 or 60 °C can be

Table 2
Comparison between desiccant drying, freeze drying, "détente instantanée contrôlée" and vacuum drying industrial processes, carried out by the Fruitis-Agritech company. +++: very good; ++: good; +: fair.

Characteristics	Desiccant drying	Freeze drying	"Détente Instantanée Contrôlée "	Vacuum drying
Powder organoleptic quality	+ + +	+ +	+	+
Powder taste similarity to the original product	++	+	+	+
Energy consumption	6 kW kg^{-1}	15– 20 kW kg^{1}	3– 4 kW kg^{-1}	5 kW kg^{-1}
Equipment cost and production capacity	2000 k€ for 5 kg h^{-1}	2000 k€ for 100 kg h^{-1}	3500 k€ for 200 kg h^{-1}	2000 k€ for 100 kg h^{-1}
Ease of implementation	+ +	+ +	+ +	+ +
Continuous processing	no	no	yes	no
Addition of chemicals (for powder consistency)	No need to add any chemical	Need to add maltodextrin	No need to add any chemical	Need to add maltodextrin

considered as an optimal compromise for the quality of dehydrated fruits/vegetables: in these temperature conditions, acceptable color and nutrient retention (for instance, ascorbic acid and phenolic content; Harbourne et al., 2009), relatively short drying time (Mrad et al., 2012) and limited structure damages are achieved (Russo et al., 2013). Certain authors have suggested the use of air temperature below the glass transition temperature of the material to be dried in order to considerably limit the degree of shrinkage (Karathanos et al., 1993; Kerdpiboon et al., 2007). On the other hand, high values of air velocities (from 1 to 2 m/s for instance) permit to both shorten the drying time and limit the extent of shrinkage (Ratti, 1994; Khraisheh et al., 1997; Zhang et al., 2014). Furthermore, decreasing the relative humidity of the drying air enables significant decrease in drying time (around 25%) and shrinkage level (Lang and Sokhansanj, 1993; Kaya and Aydin, 2009).

The slice thickness of the sample to be dried also appears as a main factor influencing the drying process: in fact, the thinner the slice, the shorter the drying time and the lesser energy consumption. An optimal thickness of 2–6 mm was thus reported (Zhang et al., 2014). Furthermore, the application of some pretreatments to vegetables or fruits prior to HAD, such as electric fields, blanching or OD, has been shown to be a promising method for the improvement of both drying rates and final product quality, with notably limited shrinkage and enhanced phytochemical retention (Pan et al., 2003; Rossi et al., 2003; Riva et al., 2005; Aktas et al., 2007; Leeratanarak et al., 2006; Sablani, 2006; Sablani et al., 2011). Moreover, applying intermittent drying using time-varying drying conditions, temperature or air velocity schemes for example, reveals as an effective strategy to shorten drying times and improve product quality (Pan et al., 1999a,b; Chua and Chou, 2003).

The quality of a freeze-dried vegetable/fruit is closely related to the size of ice crystals formed during drying, which in turn largely depends on the freezing rate and the final temperature in the freezing process. Controlling the freezing rate and the formation of small ice crystals during FD is hence critical to minimize tissue damages. The application of lower freezing temperatures and appropriate vacuum pressure (for instance, 3 mbar in order to obtain an initial temperature below or near the glass transition temperature of the material to be dried) permits rapid freezing and limited shrinkage (Anglea et al., 1993; Krokida et al., 1998; Oikonomopoulou and Krokida, 2013). It is important to remind that rapid freezing produces small intracellular ice crystals, whereas slow freezing forms large ice crystals that are able to damage cell walls (Xu et al., 2009). Applying atmospheric pressure instead of the partial vacuum FD should be avoided since threats of product structural collapses are considerable (Lombrana et al., 1997). In addition to the freezing rate, the application of alternating current electric field or low-power ultrasounds has been proposed to control the formation of small ice crystals (Xu et al., 2009; Woo and Mujumdar, 2010).

In VD, increasing drying temperatures or applying a pretreatment prior to drying (such as blanching, freezing or OD) accelerates the VD process. Shrinkage phenomena can be limited by using low vacuum pressures (Cui et al., 2005; Arevalo-Pinedo and Murr, 2005, 2007; Giri and Prasad, 2007; Wu et al., 2007; Oikonomopoulou and Krokida, 2013).

The non-uniformity of the electromagnetic field, a drawback of MW drying, can be partially offset by using wave guides and rotating tray (Cohen and Yang, 1995; Zhang et al., 2006). Limiting the applied power at 500 W might prevent overheating and the irreversible MW-drying result of scorching (Gunasekaran, 1999; Zhang et al., 2006). Finally, opting for a MW power of 915 MHz or RF heating permits to overcome one of the major MWD drawbacks: the small penetration depth of the MW field into the product (Wang and Chen, 2003).

Finally, mass transfer process during OD can be enhanced by agitation or circulation of the hypertonic solution around the sample, by increasing temperature or osmotic treatment time or by application of ultrasounds (Rodrigues et al., 2009; Bekele and Ramaswamy, 2010).

3. Effect of dry grinding process on overall quality of fruit and vegetable powders

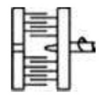
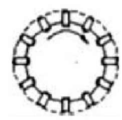
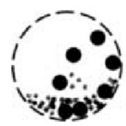
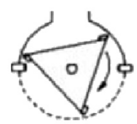
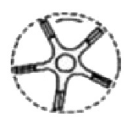
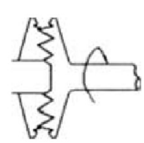
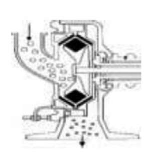
First of all, it appears important to underline the limited information existing in the literature on the effect of grinding processes on the overall quality of fruit and vegetable powders. Grinding process is another age-old known and complex process widely used in the food industry. It is a process of size reduction of solid particles subjected to mechanical forces wherein the fracture occurs within the failure of internal molecular binding forces regarding external forces. The energy required for size reduction is a direct function of particle fineness or created surface area (Murthy et al., 1999). Grinding processes, i.e. routine grinding, micronization and cryogenic grinding, have been applied to the field of food to process powders and micro-powders (i.e. of particle size less than 10 μm) raw materials (Wu et al., 2007; Zhao et al., 2009; Guo et al., 2012). The result of a grinding operation largely depends upon the food material nature and properties on one hand, and the applied technology, the distribution and intensity of the applied stress by the grinding tool on the other hand. The double heterogeneity of the source material and technology leads hence to the production of powders with different characteristics in terms of size, shape, structure, composition and functional properties detailed in the following subsections.

3.1. Conventional grinding

The dry size reduction of vegetables and fruits can be achieved in one or more of the following way: compression, impact (the most commonly used), attrition or cutting, using various equipments, such as crashers, grinders, mills, disintegrators, cutters, shredders and mincers for comminution (Velasquez et al., 2002; Peukert, 2004; Schweiggert et al., 2007). Depending on the nature of the source material and the desired characteristics of the final product, the size reduction operation can be hence performed via a specific device: knive, ball, pin, disc or hammer mill (Balasubramanian et al., 2012). Table 3 represents some of the main types of grinding devices used through conventional grinding (also known as routine grinding) of dried fruits and vegetables, as well as their schematic representation, size reduction mechanism and particle velocities.

Conventional grinding method suffer from various disadvantages like: (i) gumming of grinder walls and sieves resulting in frequent stoppage of mill for cleaning, (ii) enormous energy consumption (iii) and most importantly, the non-suitability of routine grinding for heat-sensitive materials (Ramesh et al., 2001; Indira and Bhattacharya, 2006; Zhao et al., 2009; Zhang et al., 2012). In fact, during conventional grinding, temperature rise to over 90 °C can occur (temperature rises up to 95 °C have even been reported) due to the friction-induced heat during particles fracture into smaller sizes. This local temperature increase causes important losses of aroma, nutrients and flavor components, as well as considerable quality degradation (Pesek et al., 1985; Singh and Goswani, 1999; Murthy et al., 1999). These authors have underlined the fact that about 99% of the input energy to the grinder is converted into heat whereas only 1% is used for actual size reduction. Several authors have further described the poor quality of conventional ground powders in terms of uneven particle size,

Table 3
Conventional grinding devices (adapted from Balasubramanian et al., 2012).

Type	Schematic representation	Size reduction mechanism and particle velocities
Pin and disc mill		Impact 80–160 m s^{-1}
Hammer mill		Impact 40–50 m s^{-1}
Ball mill		Impact and shear (particle velocities were not reported)
Cutting granulator		Impact and shear 5–18 m s^{-1}
Wing beater mill		Impact and shear 50–70 m s^{-1}
Knife mill granulator		Cutting 5–20 m s^{-1}
Vertical toothed mill		Shear 4–8 m s^{-1}
Turbo mill		Impact, shear and cutting 80–100 m s^{-1}

color (dark powder) and aroma (Goswami and Singh, 2003; Saxena et al., 2013). Goswami and Singh (2003) have quantified the loss of volatile oils around 30%. From their part, Zhang et al. (2009) related the significant loss of flavonoids, the reduction in radical scavenging capacity and the reducing power, as seen in koehne fruit powders for example.

The loss of valuable compounds, volatiles oils, aroma and flavor can be reduced to a certain extent either by pre-chilling (i.e., pre-cooling) or cooling the feed material during grinding by means of air blown through the grinders or with jacketed water-cooled units (Bera et al., 2001; Singh and Goswami, 2000; Murthy and Battacharya, 2008). It worth noting, however, that this latter solution seems not sufficient to significantly reduce temperature rise of the product (Saxena et al., 2013).

3.2. Micronization

Micronization is a term used to describe size reduction when the resulting particle size is less than 10 μm. Micronization size reduction involves acceleration of particles so that grinding occurs by particle-to-particle impact or impact against a solid surface. It should be noted that particle velocities in jet mills are in the range of 300–500 m s^{-1} compared to 50–150 m s^{-1} in mechanical impact mills (as shown in Table 3) (Zhang et al., 2012). Fluid-energy mills, spiral jet mill (also known as "pancake mill") and fluidized-bed mill are usually used for fruits/vegetables micronization. Nevertheless, the spiral jet mill is gradually yielded to the next generation of higher technology fluidized-bed jet mills. Even though micronization techniques are relatively more expensive and require higher energy inputs compared to conventional grinding, the outcomes for products quality are undeniable (Zhao et al., 2009). Indeed, during micronization, the surface of ground powder undergoes some changes, bringing out advantageous properties not shown in raw particles. Owing to these favorable characteristics, micronized powders might find more applications than conventionally-ground materials in the food industry for the formulation of instant and convenient foods or in other fields (Zhao et al., 2009; Huang et al., 2009; Zhang et al., 2012; Zhu et al., 2012). Kuang et al. (2011) have shown for instance, the potential use of micronized cloves and cinnamon powders as antimicrobial agents inhibiting the growth of meat spoilage organisms. For many authors, micronization appears as a useful tool for making powders with great surface properties, powder dispersibility and solubility. Chau et al. (2007) and Wang et al. (2009) have reported the significant reduction in bulk density and the great porosity values observed in micronized powders. From their part, Zhang et al. (2005a,b), Chau et al. (2007) and Zhao et al. (2009) have described an improvement of physicochemical properties, including water holding capacity, swelling capacity and water solubility index, in micronized powders. Other authors have reported the great powder flowability exhibited by micronized powders, as observed in ginger, yam starch, coconut and mushroom powders for example (Riley et al., 2008; Zhao et al., 2010; Zhang et al., 2012). Moreover, several studies have shown the good antioxidant activity and scavenging capacity displayed by micronized powders concomitant with the high total phenolics, flavonoids, carotenoids and ascorbic acid contents, as observed for prickly pear seeds, wheat barn and green tea powders (Hu et al., 2012; Rosa et al., 2012; Chaalal et al., 2013).

3.3. Cryogenic grinding

Cryogenic grinding (also known as freezer mill) performs better than any other method (Murthy and Bhattacharya, 2008) given that liquid nitrogen (at −195.6 °C, just below the vaporization temperature of liquid nitrogen at atmospheric pressure) provides the refrigeration needed to precool the feed material and maintain the desired low temperature by absorbing the heat generated during grinding (Singh and Goswami, 2000; Murthy and Battacharya, 2008). Several authors have actually reported that cryogenic milling (essentially carried out on herbal and edible spices) was better than conventional grinding in terms of retention of volatiles and flavoring components, color and particle size distribution of final powder (Pesek et al., 1985; Murthy et al., 1999; Singh and Goswami, 1999, 2000; Sharma et al., 2014; Liu et al., 2013). Balasubramanian et al. (2012) have even reported an increase in volatile oils and flavor strength (up to two-fold) in cryogenically-ground spices. Finally, Saxena et al. (2013) have reported the increase in total flavonoid and phenolic compounds in cryogenically-ground coriander seeds.

4. Alternation of drying and grinding process in powder technology

In a recent study, Djantou et al. (2011) have related the effectiveness of the alternation of drying and grinding (ADG) technique, where the material to be converted in powder form is alternatively dried and ground, for the production of fruit powder. Indeed, on one hand, the drying step permits to control the grinding adverse factors (namely, moisture) and improves the overall grinding ability; on the other hand, grinding from its part improves the drying efficiency by increasing the material specific surface, which enhances water evaporation during the second stage of drying (corresponding to weakly-bound water molecules found in the material core). Thus, compared to ordinary HAD (moisture content 17–22%) the moisture content is lower in ADG technology (2–8%), leading to harder materials easier to grind. The improved grinding abilities were primary characterized by the significant increase in grinding yield (up to 29%) resulting from reduction of grinder fouling, and the decrease in final particle size (up to 24%) and energy consumption (up to 27%) (Djantou et al., 2011).

5. Conclusion

In conclusion, among drying technologies, convective drying remains the most adopted technique in the food industries. Nevertheless, microwave drying and vacuum drying are among the alternative drying methods gaining popularity in recent years and offering great compromises between energy consumption and product quality. The controlled sudden decompression to vacuum appears as one of the most interesting novel method for implementation at industrial scale. Hybrid drying (combination of different drying methods) contributes to the dehydration process by decreasing drying time, increasing energy efficiency or improving product quality. Among routine grinding technologies and micronization, cryogenic grinding seems to be the best in terms of color, volatiles and flavor retention, narowness of particle size distribution, and energy consumption. Controlling some of the main processing (external) parameters such as temperature, pressure, air velocity, electric field strengths, etc., allows higher energy efficiencies, as well as better products quality both in drying and grinding processes.

Finally, the ADG technique can be considered as a novel approach to produce fruit and vegetable powders of superior quality.

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Effects of drying methods on bioactive compounds of vegetables and correlation between bioactive compounds and their antioxidants

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Abstract: Freeze- and heat-drying methods were applied to dry fresh vegetables (carrot, taro, tomato, red beetroot and eggplant) grown in Vietnam and then the total phenolic and flavonoid compounds extracted by alcoholic and alkaline-hydrolysis methods were evaluated to determine the effects of the drying methods on the bioactive compounds of the vegetables. Furthermore, the correlations between the content of bioactive compounds and their antioxidant capacity were also investigated in this study. The results show that phenolic and flavonoid compounds were mainly located in free form in the vegetables which was easily extracted by alcoholic solvent. A high temperature in the heat-drying method in sample preparation significantly reduced total free and bound phenolics, total free and bound flavonoids and their antioxidant capacity. The antioxidant capacity of the extracts highly correlated with free phenolic compounds (r^2 = 0.8936) and free flavonoid compounds (r^2 = 0.6682). In contrast, the antioxidant capacity of the extract did not correlate with the bound phenolic and flavonoid compounds (r^2 = 0.0124 and r^2 = 0.0854, respectively).

Keywords: Phenolics, flavonoids, antioxidant, fruits and vegetables

Introduction

Fruits and vegetables are rich in phenolic metabolites including tocopherols, flavonoids, phenolic acids, alkaloids, chlorophyll derivatives, or carotenoids (Hudson, 1990; Hall and Cuppett, 1997), that possess high antioxidant capacity and have significantly health benefits (McDermott, 2000). The beneficial effects derived from diets rich in fruits and vegetables is to protect against the risks for chronic angiogenic diseases, such as cardiovascular diseases, arthritis, chronic inflammation and cancers (Middleton *et al*., 2000; Saleem *et al*., 2002; Prior, 2003; Zhang *et al*., 2005; Chen *et al*., 2005). The phenolic metabolites in fruits and vegetables varies depending on the plant origins (Robards and Antolovich, 1997). Therefore, the extracts from different kinds of fruits and vegetables exhibited the different antioxidant capacity. The majority of antioxidant activity of fruits and vegetables may be derived from phenolic compounds. However, Burton and Ingold (1981) have shown that α-tocopherol is one of the most active *in vitro* chain-breaking antioxidants. In addition, carotenoids also have protective functions against oxidative damage (Krinsky, 1989).

Among the known fruits and vegetables, deep-colored fruits and vegetables have been reported to be good sources of phenolics, including flavonoids, anthocyanins and carotenoids and recognized as more healthy to human body, especially in the oriental countries (Qian *et al*., 2004; Sass-Kiss *et al*., 2005; Cieslik *et al*., 2006; Lin and Tang, 2007). Several deep-colored vegetables such as tomato (*Lycopersicon esculentum* Mill), carrot (*Daucus carrota* L.), eggplant (*Solanum melongena*), red beetroot (*Beta vulgaris*) and taro (*Colocasia esculenta*) have been reported to contain large amounts of bioactive compounds and have strong antioxidant capacity (Vinson *et al*., 1998). The antioxidant potential of tomato is derived from lycopene, ascorbic acid, phenolics, flavonoids and vitamin E, in which lycopene constitutes more than 60% of the carotenoids present (Roldan-Gutierrez and Luque de Castro, 2007). Lycopene is one of the major carotenoids in the diet of North American and European and the most important source of lycopene is tomato and its processed food products (Roldan-Gutierrez and Luque de Castro, 2007). The bioactive compounds in the eggplant fruit include phenolics, flavonoids, nasunin, ascorbic acid and vitamin A, which are antioxidants (Vinson *et al*., 1998) and possess high capacity in scavenging of superoxide free radicals and inhibition of hydroxyl radical generation by chelating ferrous iron (Kaneyuki *et al*., 1999; Noda *et al*., 2000). The antioxidant properties of carrot are derived from anthocyanins, carotenoids and phenolics, which are higher in purple carrot rather than in other coloured carrot varieties (orange, yellow and white) (Alasalvar *et al*., 2005). Betalains and anthocyanins are mutually exclusive in their natural occurrence, but phenolics and flavonoids have also been found in different beetroot materials (Stafford, 1994; Wende *et al*., 1999; Kujala *et al*., 2000), contributing to the strong antioxidant activity of beetroot feel extract (Kahkonen *et al*., 1999). Taro

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also contains a range of phenolic compounds which includes gallic acid, chlorogenic acid, (+)-catechin, (−)-epicatechin and (−)-epigallocatechin and the possible presence of proanthocyanidins and flavonols contributing to the antioxidant activity of taro extract (Agbor-Egbe and Rickard, 1990). Along with the consumption of fresh vegetables, the dried vegetable including dried powders of vegetables have been used to produce the deep-colored foods such as tomato ketchup, carrot powder, cakes, functional foods, etc. Drying methods play an important role in production of the dried vegetables and the bioactive compounds and their antioxidant capacity might be lost during drying process.

The objective of this study is to investigate the contribution of total free and bound phenolics and flavonoids to the antioxidant capacity of extracts of selected deep-coloured vegetables grown in Vietnam including tomato, eggplant, carrot, beetroot and taro. In addition, effects of drying methods, heat- and freeze-drying methods, on total contents of total free and bound phenolics and flavonoids and their antioxidant capacity of the vegetable extracts are also investigated.

Materials and Methods

Materials

Selected deep-coloured vegetables including carrot (*Daucus carrota* L.), taro (*Colocasia esculenta*), tomato (*Lycopersicon esculentum* Mill.), red beetroot (*Beta vulgaris*) and eggplant (*Solanum melongena*) used in this study were grown at the southern region of Vietnam and are commercially popular products at local super markets. All collected vegetables were fresh and matured. All chemicals including Folin-Ciocalteu's phenol reagent, 2,2-diphenyl-1-picyrylhydrazyl (DPPH), galic acid, rutin and other solvents were purchased from Sigma-Aldrich Chemical Company (Singapore).

Sample preparation

All vegetables were washed and edible parts were used. Fleshes of carrot, taro and red beetroot were cut into a cubic of 1cm^3. Tomato seeds were removed before cutting and eggplant peels were used instead of flesh. All pieces were divided into 2 groups. The group 1 was dried in an oven at 55^{o}C overnight, while group 2 was freeze-dried overnight. After drying, all pieces were milled into flour and stored in desiccators for later use.

Extraction of phenolic compounds

Free phenolic compounds in vegetable powders were extracted according to the method of Adom and Liu (2002). The powdered vegetable (0.5 g) was extracted with 10 ml of 80% chilled ethanol for 10 min with vortex mix. The suspension were then centrifuged at 1,500 × g for 10 min then the supernatant was collected. The extraction was repeated triplicate and the combined supernatant was then evaporated at 45^{o}C and then reconstituted with methanol to a final volume of 20 ml. The free phenolic acids were then stored at -4 ^{o}C in a refrigerator until later use.

The residue from the free phenolic extraction was directly hydrolyzed with 20 ml of 2N sodium hydroxide for 24 h with stirring at room temperature. The hydrolyzed solution was acidified to pH 2 with HCl 6N. This solution was then extracted 6 times with diethyl ether-ethyl acetate (1:1). The ether/ethyl acetate extracts were evaporated to dryness and the bound phenolic acids were dissolved in 20 ml of methanol and stored at -4^{o}C until later use. All extractions were performed in duplicate.

Determination of total phenolic contents

Contents of free and bound phenolics in vegetables were determined using the Folin-Ciocalteu's colorimetric method as previously reported by Hung and Morita (2008). Extracted solution (0.5 ml) was put into a test tube and the Folin-Ciocalteu's phenolic reagent (0.5 ml) was added. The content was vortex mixed and added with 1 ml of saturated sodium carbonate solution, followed by adjusting the volume to 10 ml with distilled water. The mixtures in the tubes were thoroughly mixed by vortexing. Tubes were allowed to stand at ambient temperature for 10 min until the characteristic blue color developed. The control was prepared in same way but the extracted solution was replaced by methanol (0.5 ml). Absorbance of the clear supernatants was measured at 725 nm using a spectrophotometer (UVD-2960, Labomed, Inc.). Galic acid was used as a standard and total phenolic content were calculated and expressed as µg galic acid equivalent (GAE) per g sample. All analyses were performed in triplicate.

Determination of total flavonoid contents

Flavonoid contents of free and bound phenolics in vegetables were determined using the aluminum chloride colorimetric method of Chang *et al*. (2002). An appropriate dilution of extract (0.5 ml) was mixed with 1.5 ml of 95% ethanol, followed by 0.1 ml of 10% aluminum chloride, 0.1 ml of 1 M potassium acetate and 2.8 ml distilled water. After incubation at room temperature for 30 min, the absorbance of the reaction mixture was recorded at 415 nm. Rutin was used as a standard and total flavonoid contents were calculated and expressed as µg rutin equivalent (RE) per g sample. All analyses were performed in duplicate.

DPPH scavenging capacity

DPPH free radical scavenging capacities of vegetable extracts were determined by the reduction of reaction color between DPPH solution and sample extracts according to the method of Huang *et al*. (2005) with minor modifications. The concentration of DPPH solution used was 0.1 mM. Mixture of DPPH solution (3.9 ml) and extracted sample (0.1 ml) was kept in the dark at the ambient temperature. Absorbance of mixtures was recorded at 525 nm for exactly 30 min. The control was made from 3.9 ml of DPPH solution and 0.1 ml of methanol and then measured at t = 0.

The DPPH scavenging was calculated according to the following equation:

$$\%\ DPPH\ scavenging = \frac{Abs(t=0) - Abs(t=30)}{Abs(t=0)} \times 100$$

Where: Abs(t=0) is absorbance of DPPH radical and methanol at t = 0 and Abs(t=30) is absorbance of DPPH radical and extracts at t = 30.

Statistical analysis

All analyses were performed in triplicate. Analysis of variance (ANOVA) was performed using Duncan's multiple-range test to compare treatment means at P < 0.05 using SPSS software version 16 (SPSS Inc., USA).

Results and Discussion

Total Phenolic Content (TPC)

Total phenolic contents of free and bound phenolic extracts from the vegetables by freeze- and heat-drying methods are shown in Table 1. Total phenolics contents of free phenolic extracts of each kind of vegetables in both drying methods were significantly higher than those of the bound phenolic extracts. These results indicate that the phenolic compounds in vegetables existed primarily in free form rather than in bound form and solvent extraction methods were simple and effective methods for phenolic recovery. By freeze-drying, free phenolic contents in vegetables were increased in order: carrot < tomato < taro < beetroot < eggplant, in which eggplant contained the highest amount of free phenolic compound (454.5 µg GAE/g db sample) and carrot had the lowest (36.6 µg GAE/g db sample). Bound phenolic contents of the selected vegetables ranged from 3.3 to 19.2 µg GAE/g db sample. The highest level of bound phenolics was also found in eggplant (19.2 µg GAE/g db sample), whereas the lowest bound phenolics was in taro (3.3 µg GAE/g db sample). Lin and Tang (2007) reported that the total phenolic and flavonoid contents in fruits and vegetables varied considerably and direct determination of each fruit and vegetables is a practical method to screen phenolic-rich fruits and vegetables but not only colors. Therefore, the results in this study indicate that eggplant and beetroot are two vegetables which are good sources of phenolics.

Table 1. Total phenolic compounds of several vegetables with different drying methods

Samples	Total phenolic compounds (µg GAE/ g sample, db)			
	Freeze-Drying		Heat-Drying	
	Alcoholic extract	Alkaline extract	Alcoholic extract	Alkaline extract
Carrot	36.6 ± 0.5a	6.7 ± 0.9b	33.1 ± 0.5c	4.6 ± 0.5a
Taro	93.5 ± 0.9c	3.3 ± 0.7a	8.6 ± 0.5a	5.8 ± 0.7a
Tomato	61.2 ± 0.7b	11.1 ± 0.7c	21.6 ± 1.0b	19.4 ± 0.5b
Beetroot	229.2 ± 1.6d	17.8 ± 0.7d	31.4 ± 0.8c	24.3 ± 1.4c
Eggplant	454.5 ± 0.5e	19.2 ± 0.7d	49.2 ± 0.7d	26.1 ± 0.2c

[a] All values are means of two extractions.
[b] The same letter in the same column is not significant different (p<0.05).

Table 1 also shows the loss of free phenolic compounds in the vegetable during drying. Total phenolic contents in free phenolic extracts found in freeze-drying vegetables were significantly higher than those in heat-drying vegetables. Total free phenolics of freeze- and heat-drying eggplant, for instance, were 454.5 and 49.2 µg GAE/g db sample, respectively. In beetroot extract, it was nearly 13 times different (229.2 and 17.8 µg GAE/g db sample) in comparison between freeze- and heat-drying vegetables. Although free phenolics were significantly higher in freeze-drying vegetables, their bound phenolics were slightly lower as compared with the heat-drying vegetables. Thus, the bound phenolics were not affected by drying methods because of their association with cell wall of vegetables. Drying is an important method of food preservation, which provides longer shelf-life, lighter weight for transportation and smaller space for storage. However, this study indicates that drying vegetables by heat which has been popularly used in food processing reduces significantly amounts of free phenolic compounds resulting in lowering the health benefit of vegetables.

Total flavonoid content (TFC)

Total flavonoid contents of free and bound phenolic extracts from freeze- and heat-drying vegetables are listed in Table 2. In free form, flavonoid contents of carrot, taro, tomato, beetroot and eggplant dried by freeze-drying method were 11.2, 7.1, 14.7, 15.3 and 42.1 µg RE/g db sample, respectively. As a result, eggplant was found to contain the highest level of free flavonoid (42.1 μg RE/g db sample) and taro was the lowest (7.1 μg RE/g db sample). The total flavonoid contents of free phenolic extracts were not quite different in both drying method. The significant reduction was account for beetroot flavonoid which were about 1.7 times reduction. Flavonoids, in bound form, of freeze-drying vegetables ranged from 3.57 to 5.41 μg RE/g db sample, whereas those of heat-drying vegetables ranged from 2.7 to 6.3 μg RE/g db sample. Similar to the total phenolic contents, the flavonoid content of the free phenolic extracts was higher than that of the bound phenolic extracts. The high amount of free flavonoid compared to the bound flavonoid was different from other investigations in grains such as rice, wheat, corn and oat (Adom and Liu, 2002; Hung *et al*., 2011). Oomah and Mazza (1996) reported that the quantity of flavonoid varied depending on the cultivar and environment effects. Thus, in this study, the highest level of total flavonoid contents was found in eggplant (46.4 and 41.5 in freeze- and heat-drying eggplant, respectively), while the lowest was in taro (10.7 and 10.9 in freeze- and heat-drying taro, respectively). The results indicate that eggplant feel extract is a good source of phenolic and flavonoid compounds and has more benefits to human health.

Table 2. Total flavonoid compounds of several vegetables with different drying methods

Samples	Total flavonoid compounds (μg RE/ g sample, db)			
	Freeze-Drying		Heat-Drying	
	Alcoholic extract	Alkaline extract	Alcoholic extract	Alkaline extract
Carrot	11.2 ± 0.2b	5.4 ± 0.2d	8.9 ± 0.2b	5.6 ± 0.1c
Taro	7.1 ± 0.5a	3.6 ± 0.2a	6.2 ± 0.3a	4.7 ± 0.2b
Tomato	14.7 ± 0.2c	4.9 ± 0.2c	15.1 ± 0.1c	6.3 ± 0.4d
Beetroot	15.3 ± 0.9c	4.6 ± 0.1bc	9.1 ± 0.1b	4.9 ± 0.1b
Eggplant	42.1 ± 0.2d	4.3 ± 0.2b	38.8 ± 0.1d	2.7 ± 0.2a

[a] All values are means of two extractions.
[b] The same letter in the same column is not significant different (p<0,05).

DPPH radical scavenging of free and bound phenolic compounds

Scavenging of the stable DPPH radical was widely used to evaluate antioxidant activity of phenolic compounds extracted from fruit and vegetables, cereal, grain, wine, etc. In this study, the antioxidant activities of free and bound phenolic extracts of the selected vegetables were evaluated using DPPH assay. DPPH scavengings of free and bound forms of phenolic extracts from the freeze- and heat-drying vegetables are shown in Table 3. DPPH radical scavenging of free phenolic extracts was highest in eggplant (92.3%) and the lowest in carrot (12.6%). The highest level of DPPH radical scavenging of the bound phenolic extracts was 6.9% in eggplant, whereas tomato was the lowest (1.3%). DPPH scavenging of free phenolic extracts from the heat-drying vegetables was also high even though the amounts of free phenolic compounds reduced significantly as affected by heat-drying method. DPPH radical scavenging of bound phenolic extracts was significant lower than that of free phenolic extracts of both freeze- and heat-drying vegetables. Thus, the free phenolic extracts of vegetables exhibited the significant antioxidant capacity which contributes to human health benefits.

Table 3. DPPH radical scavenging (%) of phenolic extracts of several vegetables with different drying methods

Samples	DPPH radical scavenging (%)			
	Freeze-Drying		Heat-Drying	
	Alcoholic extract	Alkaline extract	Alcoholic extract	Alkaline extract
Carrot	12.6 ± 0.3a	5.5 ± 0.3c	6.7 ± 0.3a	2.4 ± 0.1b
Taro	48.5 ± 0.1c	5.3 ± 0.1c	30.6 ± 1.3c	2.1 ± 0.3b
Tomato	26.0 ± 0.3b	1.3 ± 0.1a	24.0 ± 1.6b	1.3 ± 0.2a
Beetroot	50 ± 2.7b	4.2 ± 0.6b	46.1 ± 3.3d	5.5 ± 0.1c
Eggplant	92.3 ± 0.5d	6.9 ± 0.3d	86.9 ± 0.1e	2.3 ± 0.1b

[a] All values are means of two extractions.
[b] The same letter in the same column is not significant different (p<0.05).

Correlation between DPPH radical scavenging and TPC and TFC in vegetables

The high correlation between free phenolics and DPPH scavenging in vegetables is shown in Figure 1A with $r^2 = 0.8936$ whereas no correlation between bound phenolics and DPPH scavenging is observed with $r^2 = 0.0124$ (Figure 1B). These results indicate that antioxidant properties were strongly correlated to the concentration of free phenolics extracted from vegetables rather than bound phenolics concentration.

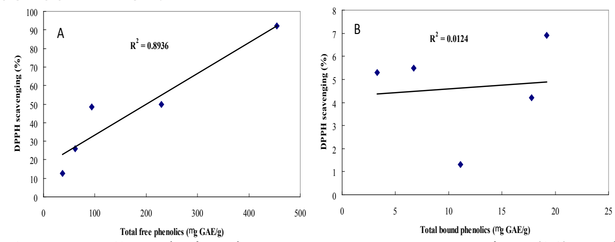

Figure 1. Correlation between DPPH scavenging (%) and total free (A) and bound (B) phenolic extracts

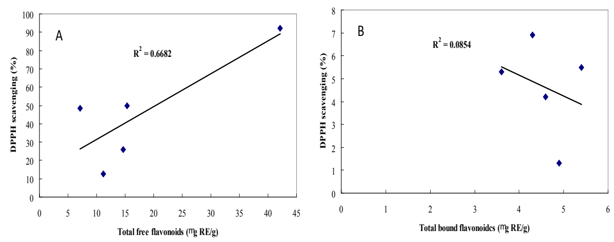

Figure 2. Correlation between DPPH scavenging (%) and total free (A) and bound (B) flavonoid extracts

As shown in Figure 2A, free flavonoid concentration was slightly correlated with DPPH scavenging ($r^2 = 0.6682$). However, no correlation between bound flavonoid and DPPH scavenging ($r^2 = 0.0854$) was also observed (Figure 2B). These results show that the free flavonoids in vegetables also contributed to the antioxidant property of the extracts.

Conclusions

The results of this study show that the total phenolic and flavonoid contents in vegetables were significantly higher by freeze-drying method than the conventional heat-drying method. The phenolic acids and flavonoids are mainly in free forms, which can easily extracted using ethanol or methanol solvents. As the result, in vegetables, the higher phenolic contents exhibited the higher antioxidant capacity and flavonoid compounds also contribute to the antioxidant properties of vegetable extracts

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