COMMUNAUTÉ FRANÇAISE DE BELGIQUE

UNIVERSITÉ DE LIÈGE – GEMBLOUX AGRO-BIO TECH

QUALITY EVALUATION OF PEACH CHIPS AND ANTICANCER ACTIVITY OF PECTIN EXTRACTED FROM CHIPS DEHYDRATED BY EXPLOSION PUFFING DRYING

LYU JIAN

Dissertation originale présentée en vue de l'obtention du grade de docteur en sciences agronomiques et ingénierie biologique

Promoteur: Luc WILLEMS (ULg, Belgium)

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The objectives of this research are to discriminate the overall quality level of peach and nectarine chips prepared by explosion puffing drying (EPD), determine the changes of texture and water soluble pectin (WSP) during EPD processing and study the anticancer activity of WSP on malignant mesothelioma (MM). Principle component analysis (PCA), Analytic hierarchy process (AHP), K-mean cluster and Discriminant analysis (DA) are used to distinguish the overall quality level of peach and nectarine chips and get the characteristic evaluation indicators, which of them (e. g. rehydration ratio and expansion ratio) are corresponding to texture properties of dehydrated products. Additionally, biochemical changes of the cell wall (e. g. pectin) are also related to texture changes. The investigate on the changes of texture and WSP at different stages of EPD processing in which osmotic dehydration (OD) was used as the pretreatment, show that OD with the appropriated concentration can improve the texture quality of dried products. The drying processing causes the degradation and structure modification of WSP, which can induce apoptosis in MM cells. EPD technology can be carried out as a potential pathway on modification of pectin, which may contribute to the development of a potential therapy against MM.

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My appreciation is more than words that I can say.

Lyu Jian

In Belgium

17th June, 2017

List of Abbreviations

°C	Celsius degree
µ	Micro
H	Hour
ANOVA	Analysis of variance
CSP	Chelator-soluble pectin
d.b.	Dry basis
DA	Discriminant analysis
DIC	Instant controlled pressure drop process
DM	Degree of methoxylation
EPD	Explosion puffing drying
F1	First characteristic vector
F2	Second characteristic vector
FA	Factor analysis
FBS	Fetal bovine serum
FT-IR	Fourier-transformed infrared spectroscopy
Gal-3	Galectin-3
GalA	Galactunoic acid
HG	Homogalacturonic chains
HM	Higher-ester pectin with DM higher than 50%
HPSEC	High-performance size-exclusion liquid chromatography
IRD	Infrared radiation drying
KC	K-means cluster
LF-NMR	Low-field Nuclear Magnetic Resonance

LM	Lowe-ester pectin with DM lower than 50%
Mcl-1	Myeloid cell leukemia-1
MCP	Modified citrus pectin
MESLN	Mesothelin
MHRs	Modified hairy regions
MM	Malignant mesothelioma
MP	Modified pectin
NK	Natural killer
NO	Nitric oxide
NSP	Sodium-carbonate-soluble pectin
OD	Osmotic dehydration
P13K	Phosphatidylinositol 3-kinase
PCs	Principal components
PCA	Principle component analysis
PG	Polygalacturonase
PL	Pectate lyases
PME	Pectin methylesterase
PP	Peyer’s patch
RG I	Rhamnogalacturonan I
RG II	Rhamnogalacturonan II
Rha	Rhamnose
RMSE	Root mean square error
RTKs	Receptor tyrosine kinases
SCFA	Short-chain fatty acids

T2	The spin-spin relaxation time
TAA	Tumor associated antigens
TFA	Trifluoroacetic acid
WSP	Water-soluble pectin
WT-1	Wilms tumor-1 gene product

LIST OF SYMBOLS

CV	Coefficient of variation (%)
DE	Degree of esterification (%)
Deff	Effective moisture diffusivity (m^2/s)
DR	Drying rate ($kg \cdot water/ kg\ dry\ matter^{-1} \cdot s^{-1}$)
ER	Expansion ratio (%)
MR	Moisture ratio
M_w	Molar mass (Da)
RR	Rehydration ratio (%)
SD	Standard deviation (%)

LIST OF FIGURES

TABLE OF CONTENTS

CHAPTER 1: GENERAL INTRODUCTION

1 PEACH AND NECTARINE

Peaches (*Prunus persica* L.) and nectarines (P. *persica* (L.) Batsch, var. nectarine) originated from China are the third most important temperate fruit group worldwide, after apples and pears [1]. On the basis of the separation of the stone from the flesh, peaches and nectarines can be divided into two groups: freestone and clingstone. Based on the amount of softening of the flesh that occurs during ripening, peaches and nectarines can be either of a melting or non-melting type [2]. According to the fresh color, yellow-fleshed varieties and white-fleshed varieties are distinguished. As the leading grower and producer in the world, China accounted for 55.24% of the total harvest areas and 50.54% of the total production of the world in 2013. However, China only accounted for 2.01% of the world export quantity and 1.72% of the world export value (FAOSTATA, 2013; http://faostat3.fao.org).

Peaches and nectarines are rich in polyphenol, vitamin C, carotenoids, flavonoids and anthocyanins, which contribute to the antioxidant capacity. However, peaches and nectarine have a short shelf-life potential due to fast softening and overall ripening, resulting in a limited period for commercialization before the product reached the consumers [3]. Therefore, low storage temperature is extensively used to extend the shelf-life of the fruits. However, chilling injury limits the storage life of peaches and nectarines under low temperature. Although peaches and nectarines can be consumed fresh, these fruits are also used for processing, which can improve their additional value and extend the shelf life. Drying technology is one of the most common processes used to improve food stability as it considerably decreases the water activity of the material, reduces microbiological and enzymatic activity, and minimizes physical and chemical reactions. Usually peaches and nectarines are dehydrated in halves and are used in bakery product fillings, fruit sauces, cake mixes and fruit leather. From last century, drying has been translated into a technology to preserve and process fruit and vegetable. The most common drying methods used in industries are open sun drying and hot air drying. Open sun drying depends on weather conditions, and is difficult to maintain the high quality of products. Hot 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. Unfortunately, the quality of a conventionally dried product is usually drastically reduced from that of the original food stuff [4]. Despite good quality of final dried products can be remained, freeze drying is only rarely used by the food industry because of its particularly high equipment and running costs and the loss of some quality features, such as flavor and color. Hence, it is more suitable for processing materials with high additional value. In this study, we would like to discover new artificial drying technology with lower unit processing

cost to dehydrate peaches and nectarines, extend their shelf-life and improve the quality of final products.

2 EXPLOSION PUFFING DRYING (EPD)

2.1 EXPLOSION PUFFING DRYING REACTORS

Among many fruit chips drying production technologies, explosion puffing drying (EPD) is an efficient non-fried drying technology [5]. EPD also known as instant controlled pressure drop process (DIC, French for Détente instantanée contrôlée) was developed since 1988 [6]. The technology is based on the self-evaporation of moisture contained in the interior of food, which takes place under a sudden pressure release to atmospheric pressure or to vacuum. With the development of science and technology, EPD technology has been applied on many fruits and vegetables, such as, apple [7, 8], mango [9], jujube [10, 11], strawberry [12] and carrot [13, 14]. EPD technology can contribute to a typical porous structure and a pleasant crispy, which are both important features for fruit and vegetable chips. Other favorable characteristics of puffed products are found in excellent color and flavor, fast rehydration, good storage stability, minimal storage and transportation costs and durability [15]. The EPD treatment also shows the effectiveness on microbiological decontamination, which is extremely important. Thus, EPD products normally have a longer lifetime than the fresh one because of the absence of insects, larvae and other factors [16]. The equipment of EPD is shown in Figure 1, which includes puffing chamber, vacuum chamber, vacuum pump, air compressor, control panel and steam generator.

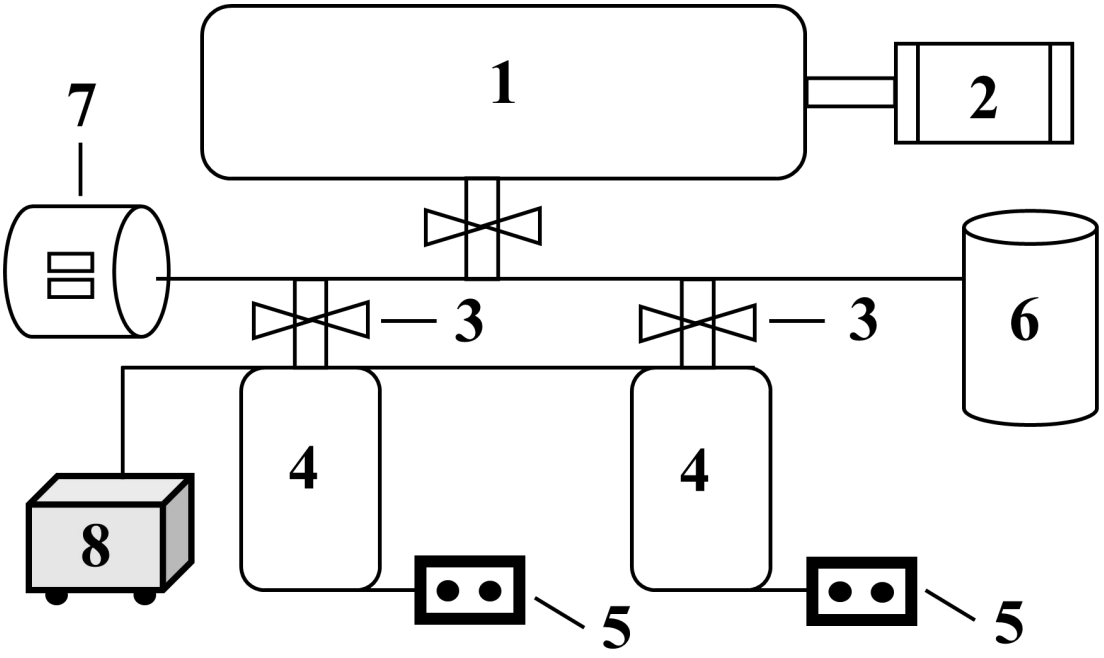

Figure 1 The diagram of explosion puffing drying equipment

1 vacuum chamber, 2 vacuum pump, 3 decompression value, 4 puffing chamber, 5 control panel, 6 water circulation equipment, 7 air compressor, 8 steam generator

2.2 STANDARD EPD TREATMENT

There are numerous parameters involved in EPD technology. These parameters vary in their importance in terms of the quality of the final products. Among the most influential parameters, there are two categories: (1) the parameters inherent to EPD technology which contribute to the definition of the technology itself such as temperature, initial and final pressures (before and after decompression), treatment time and the duration of the decompression [6]. (2) The hydro-thermo-rheological behavior of the matter, particularly its physical and rheological properties, water content and components of cell wall. Drying can cause irreversible structural damage to the cellular structure of foods. The structure of dried foods depends on the drying conditions such as temperature, relative humidity, drying time and the initial physicochemical properties of the foods. Therefore, information on the structural properties of dried food products is needed to design for the drying process.

The main steps in the EPD process are depicted in Figure 2. There is a preparing stage, in which the raw materials are washed, peeled and cut. Having carried out the foregoing preparations, blanching or other pre-treatments (if necessary), an initial partial dehydration should be carried out which is a required processing before EPD process. After that, the treated fruits will be placed in the processing vessel (in puffing chamber), in which saturated (or slight superheated) steam at pressure and temperature is supplied into the vessel for 10 or 20 mins. This stage aims to provide a homogeneous product in terms of both temperature and humidity. It is worthy to note that the duration of heat treatment by EPD and the degree of thermal degradation of nutritional ingredient can often be much lower than for other drying methods. The pressure drop occurs only when the temperature and humidity become almost homogeneous in the material through the processes of heat and mass transfer. Nowadays, the decompression is usually from high pressure or atmospheric pressure towards a vacuum. After that, the cold water injection will provide an intense cooling, while maintaining the products under vacuum. The final vacuum dehydration step is vital to bring the water content of the expanded product back down to less than 0.07 kg/kg dry basis (d. b.) to ensure storage stability.

The sudden decompression phase is regarded as the characteristic phase of EPD technology, in which a product undergoes an irreversible adiabatic transformation. This decompression induces a partial evaporation of the water within the product. The amount of generated steam is strictly related to the difference in temperature between the two stages, namely before and after the sudden decompression. The steam thus created engenders mechanical

constraints within the product, which has a viscoelastic behavior. At this stage, a complex phenomenon of product alveolation occurs. The maintenance of the product at this expanded state depends on its hardening, which is determined by the temperature and the water content of the product. Moreover, the water content acts on the amount of steam generated during the sudden decompression phase. A large amount of generated steam completely disintegrates the treated product, whereas in the opposite case the product is not well expanded. Consequently, the technology requires relatively low moisture content, in most cases around 300 g/kg [17], which means that it cannot be used alone to dry fresh food. Prior to EPD, hot air drying [12, 18] is the most commonly used pre-treatment, however, severe shrinkage and color deterioration often occur during hot air drying stage, leading to adverse effects on final product qualities, e.g. limited volume expansion. Nowadays, some novel pre-drying treatments for EPD, e.g. microwave drying [19], spray drying [20] and infrared drying [5] can be used as alternative pre-drying methods for reducing the moisture content, which can bring some positive effects on the volume expansion and texture of fruit chips.

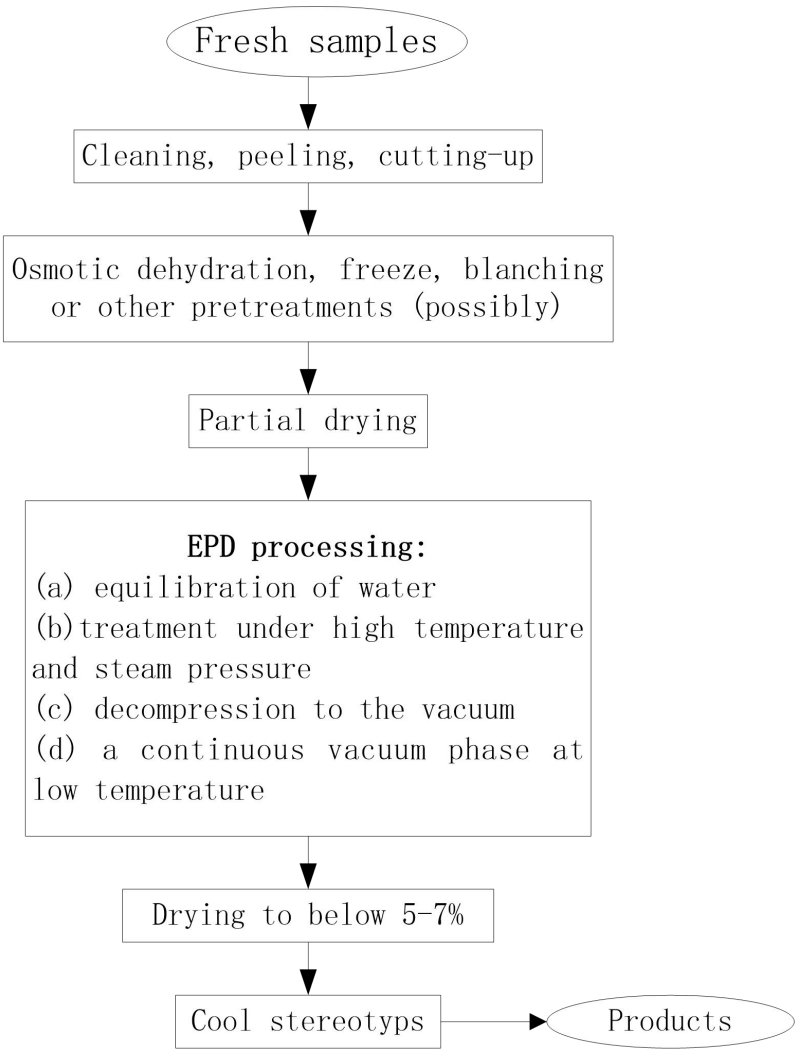

Figure 2 Schematic diagram of explosion puffing drying treatment for fruit and vegetable

2.3 PRETREATMENT PRIOR TO EPD PROCESSING

2.3.1 PRE-FREEZING TREATMENT

Freeze storage (or freeze treatment) is a promising physical treatment approach and used extensively to preserve fruits and vegetables. This preservation technology utilizes the fundamental principle of water, the density of water decreases and its volume expands when it freezes [21]. Sometimes it is inevitable that the freeze storage is required to prevent critical damage to biomass because of harsh environments (long-distance transportation or severe climate). It is accepted that fast freezing preserves local structure better. Fast freezing can deduce the production of a large number of small ice crystals that cause less migration of water, less breakage of cell walls, and consequently less texture deterioration [22].

Freeze-thawing (-40 ^{o}C – 25 ^{o}C) processes can cause an increase in pectic and hemicellulosic depolymerization because of the ice crystals formation and melting [23]. However, the fast freezing (-80 ^{o}C) is beneficial to structure preservation of fruits and vegetables based on the research on cell wall network tortuosity and anisotropy of cortical tissue [24]. Freezing at -80 ^{o}C provokes less degradation in firmness than freezing at -20 ^{o}C or immersion in liquid nitrogen [25]. The cellular structure for the freezing at -80 ^{o}C does not differ much from that of the fresh samples. After freezing at -80 ^{o}C, ice crystals in the cell appear slightly larger than the fresh samples, which make it possible to maintain cell compartments and cellular structure in the frozen state. However, thawing of the cooled tissues may lead to vacuole deterioration, giving a diffusion of free water, which may improve the drying characteristics of samples during the drying processing.

Therefore, the pre-freezing treatment (-80 ^{o}C) can be used prior to drying processing, which can deteriorate the vacuole and limit the modification of cell wall composition.

2.3.2 OSMOTIC DEHYDRATION

Osmotic dehydration (OD) has been widely applied as a pretreatment for the partial removal of water from fresh fruits and vegetables before further processing, because it can reduce energy consumption and improve product quality (e.g. color, texture, flavor and nutrients) [9]. OD pretreatment with lower concentrations than the natural cell concentration can improve the texture characteristics of products [26]. The process is typically applied on fruits such as pineapple, mango, papaya and lychee in order to enhance taste and maintain structural characteristic during drying [27].

During OD treatment, three simultaneous mass transfers occur: water transfers from the product to the solution, the solute transfers from the solution to the product; and the product's own solutes (sugars, organic acids, minerals, vitamins, etc.) leach out. The difference in osmotic pressure of the immersion solution and the product is the drying force of the process. Water and substances from the sap are transported through the semipermeable cell membrane of the biological material. The state of the membrane may change from partial to full permeability, which depends on the process conditions [28]. OD is usually performed with sucrose solution as osmotic agent, whose concentration plays an important role in OD. The higher the concentration of the sucrose solution, the higher the rate of osmosis takes place [29].

OD makes it possible to modify the composition of the raw material. Additionally, rearrangements of structure with subsequent changes in volume, density and porosity can also be induced during OD [30].

2.3.3 Infrared radiation drying

In this study, the wavelength of infrared radiation drying (IRD) is in the range of 1-4 μm which covers the maximum absorption wavelength of water molecule. As a consequence, high heating speed is one of the most obvious advantages of IRD [31]. When IRD is used to dry moist materials, it penetrates into them and the energy of radiation converts into heat [32]. Compared with conventional drying methods, IRD has gained popularity as an alternative drying method for a variety of agricultural products, such as bulgur, tomato, jujube etc [33, 34]. Notable effects of IRD on foods are that it increases porosity of banana, increases rehydration potential of onion [35], reduces overall color changes in pineapple and potatoes [36], in spite of high drying rates and firmer texture of dried blueberries [37].

IRD combined with hot air drying has a positive effect, such as reducing the drying time and specific energy consumption of agricultural materials [38]. Moreover, IRD can improve the quality of dried products, for example, decreasing the hardness of banana slices when combined with low pressure superheated steam and improving rice yield of paddy after coupled with fluidized bed drying [39]. IRD coupled with air jet impingement can decrease drying time and improve the quality of potato [40]. Therefore, IRD can be used prior to EPD processing to improve the drying characteristics and quality of peach fruits.

2.4 Quality evaluation of fruit chips dehydrated by EPD technology

EPD technology is not suitable for all the varieties of peaches and nectarines due to their different physicochemical and structural characteristics, such as high content of water, protein and carbohydrates. In this sense, the quality of peaches and nectarines chips dehydrated by EPD technology should be evaluated and the excellent varieties for

processing chips should be selected. However, most researches on quality evaluation of fruit chips are based on sensory evaluation instead of overall quality evaluation. The quality of chips includes not only sensory quality but also physicochemical, nutritional quality and processing quality. Meanwhile, no clear information is available on quality evaluation of peaches and nectarines chips and on suitable varieties for processing peaches and nectarines chips. The variety of closely related but independent quality factors increases the difficulty of a overall evaluation [41]. Therefore, it is necessary to search for methods to simplify the evaluation process. Moreover, the overall quality evaluation models should be established to select the best varieties for producing peaches and nectarines chips.

Mathematical analysis methods have been reported to evaluate the quality changes of fruits and vegetables products [42]. Principle component analysis (PCA) is a statistical procedure that uses an orthogonal transformation to convert a set of observations of possibly correlated variables into a set of values of linearly uncorrelated variables called principal components (PCs). Generally, the PCs are lower than the number of original variables [43]. Factor analysis (FA) is a statistical method used to describe variability among observed, correlated variables in terms of a potentially lower number of unobserved variables called factors. Analytic hierarchy process (AHP) is a mathematical method for analyzing complex decision problems, with analyzing complex decision problems with multiple criteria [44]. It provides a comprehensive and rational framework for representing and quantifying its elements, for relating those elements to overall goals, and for evaluating alternative solutions. Discriminant analysis (DA) is often used in statistics and pattern recognition to find a combination of features that characterizes or separates two or more classes of objects or events. DA is also a statistical analysis technique for producing score plots for the analyses [45].

As already mentioned, PCA, AHP and DA will be useful techniques to simplify the overall quality evaluation process of peach and nectarine from different varieties.

3 PECTIN

When fruit and vegetables are heat-treated during combined drying processes, water status and some physicochemical changes of the structural constituents of cell wall and intercellular tissue can be observed [46]. Pectin has been identified as a critical structure component of plant cell wall and is predominantly presented in the middle lamella [47]. The main role of the plant wall components is to give mechanical strength to plants, to maintain an extracellular water phase by imbibition and to provide a barrier from external environment [48]. Pectin belongs to a family of oligosaccharides and polysaccharides that have common features, but are extremely diverse in their fine structure [49].

3.1 CLASSIFICATION AND STRUCTURE CHARACTERISTIC OF PECTIN

The exact macromolecular structure of pectin is still under debate. However, the FAO and EU stipulate that pectin must consist of at least 65 % galacturonic acid (GalA). The backbone is comprised of α-(1-4)-D-galacturonic acid residues, forming long homogalacturonic chains (HG), which represents the smooth region. Some of the Gal residues in the smooth region are partially esterified with methanol at the C-6 carboxyl group and may also be esterified with acetyl groups at C-2 or C-3 [50], which is an important indicator for evaluating the functional properties and sources of pectin. The backbone is interrupted by the insertion of α-(1-2)-L-rhamnopyranosyl residues with a large number of neutral sugar side chains, such as rhamnose, xylogalacturonan, arabinan, arabinogalactan and galactose [51], which represents the hair region. The hair region consists of rhamnogalacturonan I (RG I) and rhamnogalacturonan II (RG II). RG-I is composed of repeating units of [α-(1-4)-GalA-α-(1-2)-Rha, which can be substituted at O-4 with mainly galactosyl- and/or arabinosyl-containing neutral sugar side chains. Whilst the backbone of RG II is made up of α-(1-4)-bound GalA residues, containing cluster of different hetero-oligomeric side chains [52].

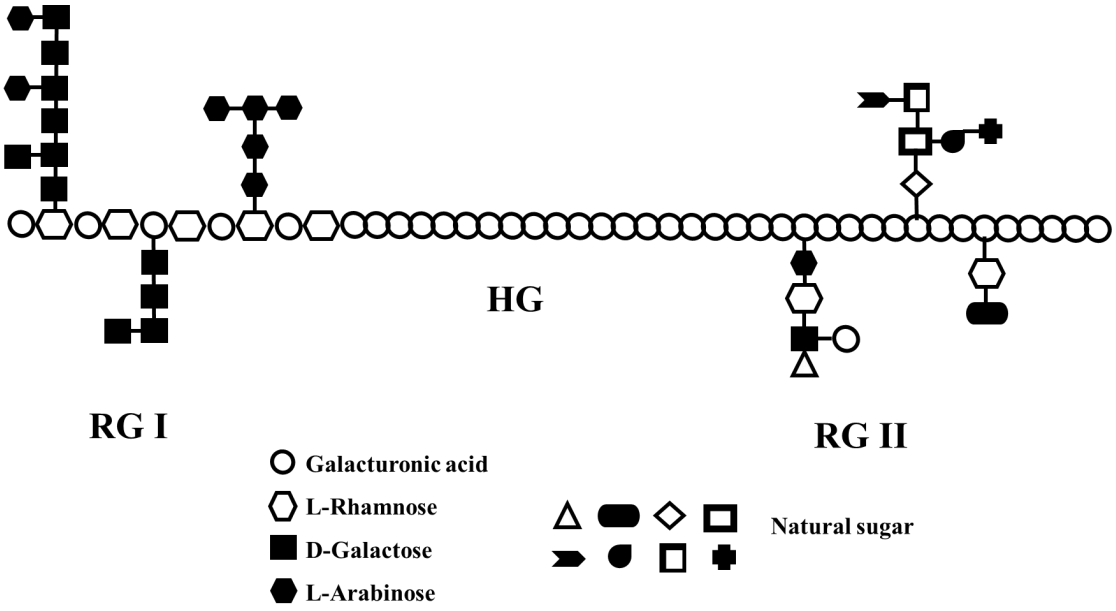

Figure 3 The basic structure of pectin

Note: HG, RGI and RGII represent homogalacturonic chains, rhamnogalacturonan I and rhamnogalacturonan II, respectively. (Figure is modified from Mohen, D. [53])

Part of the carboxylic groups in the galacturonic chain exists in the methyl ester form, and the degree of methoxylation (DM) divides pectin into two types: high-ester pectin with DM higher than 50% (HM), and low-ester pectin with DM lower than 50% (LM). Based on the solubility, pectin can be fractionated into three pectin fractions: water soluble pectin (WSP), chelator-soluble pectin (CSP) and sodium-carbonate-soluble pectin (NSP). Based on the type of modifications of the backbone chain, pectic substances are classified into protopectin,

pectic acid, pectinic acid and pectin [54]. Nowadays, more researches focus on "modified pectin (MP)", which has been broken down into small fragments. Modification can be made by chemical, enzymatic or heat treatment [55], and then pectin derivatives can be formed. With the modification, properties of pectin such as solubility, hydrophobicity, physicochemical and biological characteristics may be changed and some other new functional properties may be created [56].

3.2 PHYSICO-CHEMICAL CHARACTERIZATIONS OF PECTIN

The degree of esterification (DE), an important functional parameter of pectin, determines the degree of reactivity with calcium and other cations [57] and may have an important role in the solution behavior, gel forming ability and properties of pectin [50]. HM forms a gel under acidic conditions in the presence of high sugar concentrations, and can be used as gelling agents in fruit-based products, especially in the manufacture of jams and fruit preservatives [56]. LM may form gels in a broad pH range in the presence of multivalent cations, particularly Ca^{2+} ions. However, the gel forming properties of pectin varies widely depending on the source and the DE. Due to a block wise distribution of carboxyl groups, citrus pectin with the same DE will form gels with a slightly higher setting temperature and a more elastic texture when compared to apple pectin. The same block wise carboxyl groups' distribution of HM additionally provides advantages regarding protein stabilization in acidified milk drinks [57].

Pectin contains a complex mixture of polysaccharides and its major component consists of α-D-galacturonic acid units. In this main chain, L-rhamnose units are occasionally inserted. Other neutral sugars such as arabinose, galactose, xylose, fructose and mannose may be attached as side chains. The pectin carbohydrate composition is an important feature for the chemical characterization of these polymers and for the study of their functional or biological properties [58]. In solutions, citrus pectin is less flexible than apple pectin due to higher amount of rhamnose groups in the latter [59]. The degree of branching of the pectin chain with neutral sugar side chains is suggested to result in a more compact molecule [60]. Rhamnogalacturonans and hairy regions can limit the interactions with procyanidins, which may reduce the polysaccharide fermentation and increase the production of procyanidin colonic metabolites [61]. The interact mechanisms between procyanidins and pectic fractions is depended on the pectin side chains. In addition, alkali treatment can modify the neutral sugar side chains, namely increase the ratio of RG I and HG content of sugar beet pectin, which can enhance the bioactivity of pectin and induce apoptosis of colon cancer cells [62]. Changes in composition and content of neutral sugar will significantly affect cell wall swelling and pectin solubilization during fruit and vegetable ripening [63]. Food processing, such as high pressure homogenization, can catalyze the depolymerization of

pectin, which is dependent on its source, most likely due to the presence of neutral sugar side chains [50].

Weight-average molar mass (M_w) and its distribution refer to aggregates of pectin, which can be measured by the method of high-performance size-exclusion liquid chromatography (HPSEC). M_w and its distribution of the polysaccharide fractions (WSP, CSP and NSP) from different fruit and vegetable vary greatly. WSP extracted from broccoli and carrot has lower M_w than the pectic polysbroccoli and carrot accharides of the CSP and NSP. Furthermore, the NSP pectic polymers of both broccoli and carrot display the narrowest size distribution of the polysaccharide fractions [52]. The CSP fraction, in raw broccoli consisting of ionically cross-linked pectic polymers with a high M_w, is extended with new ionically cross-linked pectin, i.e. low M_w pectin chains previously occurring in the WSP fraction [47]. Pectin depolymerization is a common change accompanying fruit ripening, which is associated with the reduction of pectin polymer size. In the early developmental stages of peaches, CSP was presented with very high M_w and with a shoulder of midsize molecules and very low amounts of small but still polymeric molecules. This M_w profile changes substantially until the fully ripe stage is reached, when a dramatic shift in M_w profile occurs consisting of a loss of high molecular weight molecules and an accumulation of much smaller molecules. A further shift away from high molecular weight molecules occurs at the overripe stage. By contrast, the size distribution of NSP from peach does not change significantly during development, ripening or senescence [64]. Pectin, primarily the larger molecules above a specific M_w threshold, can be depolymerized during processing [50]. High hydrostatic pressure treatment can destroy the connection between the side chains of pectin extracted from sugar beet with high M_w, which leads to the decrease of the M_w [65].

3.3 Pectinolytic enzymes

There is a group of pectinolytic enzymes broadly known as pectinases that are involved in pectin degradation. The most important classes of pectinolytic enzymes are pectin methylesterase (PME), polygalacturonase (PG) and pectate lyases (PL). The main role of these enzymes is basically to degrade pectin in the middle lamella and primary cell wall of young plant cells [29].

PME is widely distributed in plants and microorganisms. In plants, PME is bound to the cell wall by electrostatic interaction. It catalyzes the de-esterification of methyl esters of polygalacturonic acid polymers to form pectinic acids/pectic acids and methanol [66]. PME activity is linked to chemical changes in cell wall-middle lamella structure occurring during the thermal treatment of the vegetal tissues, affecting their mechanical behavior. PME can be activated at temperature between 50 and 70 ^{o}C and inactivated rapidly at 80 ^{o}C [67]. However, PME is very stable under pressure treatments, which can be beneficial for structure improvement of processed fruit and vegetables by enhanced PME activity during

high-pressure processing [68]. PME action is a preliminary step in a process and yield a substrate for the action of PG, whose activity results in hydrolysis of galacturonic acid residues in the pectin polymer and subsequent loss of firmness of fruit and vegetables during industrial processing [69].

PG is an enzyme that catalyzes the cleavage of pectin chains with a low DE. In most cases, PG is presented in fruit and vegetables in two isoforms, PG1 and PG2. All of them are structurally related, but PG1 and PG2 have markedly different thermal stability. PG1 is more heat resistant, requiring temperatures of 85 ^{o}C for significant inactivation. In contrast, PG2 is readily inactivated around 65 ^{o}C [70]. However, PG1 shows similar pressure stability with PG2 in Fachin et al. study [71]. Additionally, PG2 can be converted into PG1 by a catalytic polygalacturonase subunit (so-called β-subunit) interacting with PG2, which can enhance the thermal resistance of the catalytic polypeptide of PG [72]. Therefore, a controlled activity of PME with selective PG can result in some benefits for fruits and vegetables processing.

PL can degrade pectin polymers directly by a β-elimination mechanism that results in the cleavage of α-(1-4)-D galacturonosidic linkages in HG, while other pectinases act sequentially to degrade pectin molecule completely [29]. The depolymerisation catalyzed by PL can result in the formation of a double bond between C-4 and C-5 at the newly formed non-reducing end. Additionally, PL acts by depolymerizing cell-wall polygalacturonides in the present of calcium ions, thus destroying the integrity of the plant tissues [73]. For PLs, highest specific activity is recorded using pectin with moderate DE (20-50%) [74]. PL is capable of clarifying fruit juices containing highly esterified pectin without producing methanol during clarification, which is normally found in commercial products [75]. Nowadays, PL activity and PL genes have now been detected in many plant species, often including sequences that are specifically expressed in fruits [76]. A putative PL has been identified in plants and involved in fruit and vegetable textural changes during plant development and ripening [73]. However, little information is known about the food-technological relevance of PL.

3.4 Effect of thermal processing on pectin

Processing techniques such as thermal and static high pressure are known to cause drastic effects on the structure of pectin that can result in depolymerization, demethoxylation and degradation depending on the temperature, enzymatic activity and pH [77]. The implications of food processing on the structure-related quality of plant-based foods can basically be explained by two phenomena: 1) fragmentation of cell wall pectin as a result of depolymerisation reactions, which can be catalyzed by non-enzymatic and enzymatic modifications; 2) pectin cross-linking through salt bridges (mostly calcium) [74]. Through depolymerisation of cell wall pectin, PG induces loss of structural integrity of tissue systems, which may deliberately be enabled or enhanced (for improving clarification or extraction of juice), traditionally through thermal processing. When heating pectin, especially for pectin

with high DE, splitting of glycosidic linkages between GalA residues can be taken place by a process of β-elimination, which is considered to be the major driving force behind tissue softening upon thermal processing of fruits and vegetables. The second phenomenon, salt bridge formation between HG chains, can enhance cell adhesion upon cross-linking of the middle-lamella pectin, reinforces the cell wall and increases firmness. Particularly, exogenously added ions (calcium) can be supplemented to promote network formation, through soaking or infusion [7].

During drying processing, which in most cases is a thermal treatment, the modification of pectin structure is correlated to the physicochemical properties (e.g. hardness and crispness) of the final product. Constenla et al. [78] found that temperature during apple pomace dehydration in a rotary dryer affected both the DE and the degree of polymerization, which was associated with the heat-induced reduction in M_w. The behavior of depolymerization can reduce the intrinsic viscosity of pectin extracted from apple pomace. During sun-drying process, pectin extracted from Japanese persimmon fruit is modified, which is attributable to degradation of the side-chains in hairy regions [79]. The modification of pectin in the cell wall may produce certain amounts of small molecules, which can provide an increasing osmolality to the material. The network of the cell wall can be disorganized and misaligned to a certain extent during drying process. This can damage the integrity of the primary cell wall and/or middle lamella of fruit slices, leading to a decrease in the strength of intercellular adhesion. The above mentioned modifications in pectin can affect the physicochemical properties of fruit chips. Reduction in intercellular adhesion strength may be in favor of tissue/cell separation and reduce the internal structural resistance for volume expansion during the puffing phase of EPD. This may contribute to a more porous microstructure as well as a crispier texture of chips dried by EPD. However, changes in the texture of EPD-processed peaches resulting from variations in the physico-chemical characteristics of pectin are poorly investigated. On the other hand, pectin can be modified by the drying processing, which may improve the biological and anticancer activity of pectin.

4 ANTICANCER ACTIVITY OF PECTIN

Pectin not only gives mechanical strength to plants to provide a barrier from external environment, but also plays a significant inhibitory role in cancer cell metastasis, invasion, angiogenesis and survival [80]. Pectin, especially MP, can inhibit tumor growth or cancer metastasis in different cancer cell types [81, 82, 83, 84].

4.1 ANTICANCER ACTIVITY OF NATURAL PECTIN

As a dietary fiber, pectin extracted from apple, citrus, potato or sweet potato possesses anticancer activity, although this observation is controversial. The anticancer mechanisms of neutral pectin may correlate with their probiotic activity and immune potentiation [85].

Pectin is not enzymatically digested in the small intestine but is fermented by microbial in colon into short-chain fatty acids (SCFA), such as butyrate, which can regulate apoptotic proteins in colonic crypts and enhance colonocyte growth. Apple pectin can decrease the activity of β-glucuronidase which is correlated to colon cancer development. Apple pectin has the potential to induce apoptosis in prostate cancer cells through increasing the release of nitric oxide (NO), which may have pro-apoptotic as well as anti-apoptotic effects [82]. Ginseng pectin has also been shown to inhibit the actions of galectin-3 (Gal-3), a β-galactoside-binding protein associated with cancer progression [86]. Although neutral pectin is mainly active within the gastrointestinal tract, evidences suggest that pectin can stimulate the immune system. Oral administration of pectin isolated from Korean persimmon vinegar not only increases IgA production via transforming growth factor TGF-β and interleukin IL-6 but also stimulates hematopoietic growth factors [87]. In experimental lung metastasis studies of Colon 26-M3.1 carcinoma cells, pectin purified from peels of Korean Citrus Hallabong significantly enhances production of IL-6 and IL-12 by murine peritoneal macrophages. Pectin also inhibits tumor metastasis via activation of macrophages and natural killer (NK) cells [88]. Pectin from *Centella asiatica* (L.) Urban, a traditional herbal compound founded in Asia, increases immunological activity of T and B cells, which is modulated by the carboxyl and acetyl groups of this type of pectin [89]. Pectin is also capable of diminishing blood cholesterol levels and of stimulating lipid excretion. The exact mechanisms underlying these effects are not known yet [48].

4.2 Anticancer activity of modified pectin

New and exciting researches are now being carried out on the effects of what is ambiguously called ‘modified pectin’ (MP). Modification via pH, temperature, pressure, and/or enzymatic degradation produces MP that is much lower in molecular weight and less structurally complex, compared with the original pectin. MP is believed to be better absorbed and utilized by the body than ordinary, long-chain pectin [90]. Several in vitro studies have shown that various forms of MP have antitumor properties [91].

4.2.1 MP and cancer cell apoptosis

Although MP is not capable to prevent 100% metastatic tumor formation, it can induce cell apoptosis. In the case of human prostate cancer, citrus pectin modified by heat treatment, induces significant apoptosis levels of androgen-responsive and androgen-insensitive cells comparable to pH-modified CP [92]. Nagel et al. [93] have demonstrated that treatment of Swiss 3T3 albino mouse embryo fibroblasts with pectin modified hairy regions (MHRs) dramatically decreases their proliferation index. Treatment of MHRs increases the percentage of cells in sub-G1 phage, decreases cells in S phage and activated the interaction between pectin and serum-adhesive protein. These evidences prove that MP possessed antitumor potential by inducing cancer cell apoptosis.

4.2.2 Molecular interaction of MP in cancer

Various investigations indicated that MP can bind to the carbohydrate recognition domain on the pro-metastatic protein Gal-3, which is widely expressed in mammalian cells and involved in various biological processes, such as cell migration, proliferation, differentiation, apoptosis, as well as cell-cell and cell-matrix adhesion [94]. This binding inhibits Gal-3's ability to promote cell adhesion and migration [95]. The RG-I region carrying very short galactan side chains of okra pectin, can inhibit the proliferation of melanoma cells and induce apoptosis, probably by interacting with Gal-3 protein [96]. Modified citrus pectin (MCP) has a strong effect on cell viability on HaCaT cells due to the galactose side chains, which may be masked and exposed in MCP, thereby allowing MCP to bind to their appropriate receptors (e.g. Gal-3) on HaCaT cells [97]. In ovarian cancer, PectaSol-C MCP can decrease basal caspase-3 activity and inhibit the function of Gal-3, which may sensitize ovarian cancer cells to chemotherapeutic drugs (e.g. parlitaxel) [98]. Nangia-Makker et al. [99] have demonstrated that oral intake of MCP in NCR nu/nu mice injected with MDA-MB-435 cells inhibits carbohydrate-mediated cancer growth, angiogenesis and metastasis *in vivo*, by blocking the association of Gal-3 to its receptors. These studies revealed that MP inactivates key transcriptional factors, cell cycle regulators and cancer metastatic factor galectin-3, which can lead to the inhibition of cancer cell growth, angiogenesis and metastasis.

4.2.3 Malignant mesothelioma (MM)

Malignant mesothelioma (MM) associated with asbestos exposure is a fatal tumor that is resistant to various treatment options available at present. Although the use of asbestos has decreased in recent years, large numbers of new MM patients are still diagnosed each year because of the long latency period (up to 40 years) of MM. Three histologies are usually identified in MM: epithelioid, which are the most common, biphasic and sarcomatoid [100]. In contrast to other solid malignancies whose aggressiveness is exerted locoregionally, MM morbidity results from regional invasion [101]. For most patients, MM is diagnosed at late stages. With average survival times ranging from a few months to less than 1 years, MM has poor prognosis. The current standard therapy for MM consists of surgical resection, combination chemotherapy with cisplatin and pemetrexed, and potentially radiation [102]. Takayama et al. [103] have investigated inhibition of PAI-1 (SERPINE1), whose expression has been associated with poor prognosis in MM patients. Systemic administration of SK-216, a specific inhibitor of PAI-1, can suppress tumor progression of MM cells *in vivo* through inhibition of angiogenesis regardless of which angiogenic factors are produced by MM. Furthermore, the combination of SK-216 and cisplatin is shown to enhance the antitumor effect of bevacizumab and cisplatin.

Although asbestos exposure is the main risk factor in the development of mesothelioma, the molecular mechanisms remain unclear. Khodayari et al. [104] have demonstrated the mechanism of ephrin-A1-mediated tumor suppressive and apoptosis induction signaling in MM cells. Their experiment show that ephrin-A1 treatment upregulated miR-302b expression in MM cells and attenuated cell proliferation and tumor sphere formation via repression of myeloid cell leukemia-1 (Mcl-1). Receptor tyrosine kinases (RTKs) such as MET and its downstream target phosphatidylinositol 3-kinase (P13K) were overexpressed and activated in a majority of MMs. A dual MET/P13K targeting strategy in treating MM demonstrates superior efficacy in inhibiting multiple aspects of tumor cell growth *in vitro* (viability, migration, colony formation) and *in vivo* (a PDX mouse model) [102] .

Standard mesothelioma therapies consisting in treatment with pemetrexed and cisplatin improve survival of patients (about 3-4 months). However, due to the resistant of MM to chemotherapy and radiation, immunotherapy may be the most promising approach for MM treatment. The principle of this approach relies on the stimulation of the immune response through vaccination with tumor associated antigens (TAA). The Wilms tumor-1 gene product (WT-1) and mesothelin (MSLN) expressed by mesothelioma cells can be considered as TAA [105]. So WT-1 and MSLN may be used to induce tumor specific response [101]. Immunotherapeutic agents targeting MSLN, such as SS1P and amatuximab, are currently being evaluated in phase I and phase II clinical trials, respectively [106, 107].

Another strategy is based on antigen presentation by dendritic cells (DC) upon ex vivo stimulation with tumor lysates. DC can establish a link between innate and adaptive immunity by initiating the development of an antigen-specific immune response. Unfortunately, MM is poorly immunogenic and only a few mesothelioma-specific TAA are known to date. Proteomic analysis of exosomes released by human MM cell lines reveal that exosome is enriched with molecules that may be involved in antigen presentation. Based on the above results, Mahaweni et al. [108] have investigated the feasibility of using MM cell line-derived exosomes to load DCs in a mouse mesothelioma immunotherapy model. Their results show that the exosomes derived from non-immunogenic and highly suppressive mesothelioma tumors are enriched with tumor antigens and are capable of inducing anti-cancer responses when presented by DCs to T cells. Immunotherapy thus attempt to modulate the immune system to strengthen the anticancer effect. The undoubtedly extended research should be further carried on, which will hopefully prove to be an effective treatment for mesothelioma [105].

Pectin has shown potential as an anti-cancer agent for cancer prevention and treatment [109], hence, we can expand its application on MM therapy. However, there is no study focused on the effect of pectin on MM. Therefore, the effect of pectin on MM cell proliferation, migration and apoptosis is investigated in this thesis.

In conclusion, peach and nectarine can be consumed fresh and processed into juice, pulp and chips. Attempts have been reported on application of single drying methods of peaches and nectarines, such as microwave drying, hot air drying. There are limited reported studies on the application of a combined drying technology on peach processing. Additionally, many studies on peaches and nectarine drying always focus on the drying characteristics, while no clear information is available on overall quality evaluation of peach and nectarine chips. The research on the selection of suitable varieties for processing peach chips is also limited. The variations in the quality of the dehydrated products from different varieties are always distinguished based on sensory quality instead of overall quality. Therefore, evaluation system of the overall quality of peach and nectarine chips based on precise mathematical analysis methods is needed.

Pretreatments used prior to EPD technology, such as pre-dry treatment, freezing treatment and OD treatment make it possible to modify the composition of the raw material. Thus, attempts report on the effect of preteatments on the final texture quality of dehydrated products. Generally, EPD technology should be assisted by the pretreatments, which is named the combined drying processing. However, little available studies focus on the changes of the texture quality of dehydrated products during the combined drying processing.

Pectin structure is correlated to the physical and chemical properties (e. g. texture quality) of fruit and vegetable. Processing techniques such as thermal and static high pressure are reported to cause drastic effects on the characteristics of pectin that can result in depolymerization, demethoxylation and degradation. Drying processing (e.g. EPD treatment) also can affect the modification of pectin. However, changes of the characteristics of pectin during the combined drying processing are poorly investigated. Pectin, especially MP has potential biological activity for cancer prevention and treatment. Studies demonstrate that pectin can induce apoptosis in cells of prostate cancer, melanoma cancer, ovarian cancer and lung cancer. However, no study expands the application of pectin and MP on MM therapy.

5 OBJECTIVES OF THE STUDY

The objectives of this study are as follows:

(1) to obtain the characteristic evaluation indicators and separate the overall quality level of peach chips from different varieties,

(2) to identify effect of IR-EPD treatment assisted by OD (the combined drying processing) on the characteristic evaluation indicators, i. e. texture,

(3) to identify changes of characteristics of WSP extracted from peach slices at different stages of the combined drying processing,

(4) to identify the effect of the WSP on the apoptosis of MM cells.

To achieve the goals, the following questions will be answered:

1. Are there mathematical analysis methods to simplify the overall quality evaluation workload and obtain the characteristic evaluation indicators? Can the discriminant models be established to evaluate and predicate the overall quality of peach chips ?

2. Does OD pretreatment modify the drying characteristics of peach slices during the IRD processing? How is the change of the characteristic evaluation indicators (i. e. texture) during the combined drying processing?

3. How is the modification of the WSP and water status during the combined drying processing?

4. Does the WSP extracted from peach and peach chips induce apoptosis in MM cells? Is there a relationship between the structure of modified pectin and its anticancer activity?

To answer these questions, the following hypotheses are tested:

1. Mathematical analysis methods (i.e. PCA, AHP and DA) can be applied to reduce the data dimensionality, find the combination of features and separate the overall quality levels of peach chips.

2. In general, the partially dried material (usually the water content is 0.45-0.55 kg/kg d.b.) should be prepared prior to EPD treatment. Serious shrinkage will be observed in samples during IRD treatment. With EPD treatment, materials will be puffed and the shrinkage will be decreased or disappeared. Materials pretreated by OD with appropriate concentration will have a better final quality in terms of nutrient retention and texture quality.

3. Water status and WSP characteristics can be changed during the combined drying processing. Additionally, WSP is susceptible to enzymatic and heat-induced degradation and conformation changes.

4. Pectin can suppress tumor incidence, inhibit cancer cell metastasis and induce apoptosis in cancer cells. Therefore, WSP extracted from peach and peach chips may induce apoptosis in MM cells.

Overall, this study allows a better understanding of the application of mathematical analysis methods on quality evaluation of peach and nectarine chips prepared by EPD. The characteristic evaluation indicators are selected based on the results of the application of

mathematical analysis methods. After that, the changes of texture characteristics and WSP are investigated. At last, a great importance is given to the effect of WSP on apoptosis in MM cells.

The quality of peach chips includes sensory quality, physicochemical quality, nutritional quality and processing quality, which can be evaluated by complex factors. Different quality factors are closely related but relatively independent. Therefore, Chapter 2 and Chapter 3 search the effective methods to select the characteristic indicators, which reflect the most information on product quality of peach chips. Additionally, the scientific models are very important to evaluate and predict the overall quality of peach chips from different varieties.

CHAPTER 2: QUALITY EVALUATION OF YELLOW PEACH CHIPS PREPARED BY EXPLOSION PUFFING DRYING

This chapter is a modified version of the following research article:

Lyu, J., Zhou, L., Bi, J. Liu, X. and Wu, X. (2015) Quality evaluation of yellow peach chips prepared by explosion puffing drying. Journal of Food Science and Technology, 52(12): 8204-8211

The aim of this study was to evaluate the overall quality of yellow peach chips prepared by explosion puffing drying. Seventeen evaluation indicators were measured, including color, rehydration ratio, texture and so on. A principal component analysis was applied to characterize the evaluation indicators. A analytic hierarchy process was used to calculate the weights of the characteristic indicators. The levels in varieties were classified by K-means cluster (KC) and discriminant analysis (DA). Seventeen evaluation indicators varied widely from with a coefficient of variation ranging from 3.58% to 852.89%. Reducing sugar content, out-put ratio, water content, a value and L value were selected as the characteristic indicators with their weights, 0.0429, 0.1140, 0.4816, 1.1807 and 0.1807, respectively. The quality levels in fifteen varieties were effectively classified by discriminant functions obtained by KC and DA. Results showed that Senggelin was the best variety to process yellow peach chips, while, Goldbaby was the worst variety to process yellow peach chips.

Key words: yellow peach chips; quality evaluation; principle component analysis; analytic hierarchy process; discrimination analysis

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1 INTRODUCTION

Peach (*Prunus persica* L. Batsch) is a climacteric stone fruit species originated from China, and it can provide high nutrition and a pleasant flavor. Peaches are the fourth most important fruit group in China, after oranges, apples and pears. Peaches have a short shelf-life potential due to fast softening and overall ripening. Drying techniques can be used to preserve agricultural products, and prevent concurrent of undesirable changes induced by microbial activity. Nowadays, new and /or innovative techniques that increase drying rates and enhance product quality have achieved considerable attention [35]. There are several processing technologies for peach chips, such as deep fat frying, freeze-drying and hot air drying. Despite quick rehydration and relatively good quality of final dried products, freeze drying is only rarely used by the food industry because of its particularly high equipment and running costs. The use of freeze-drying is therefore limited to a few cases of high value products [6]. Explosion puffing drying (EPD) provides products that are less deformed, which can be more quickly rehydrated, with good preservation of flavor, for lower unit processing costs [110]. As a rapid drying technology, EPD has been used to dry some vegetables and fruits, such as apple [7], purple maize [111], jujube [11], and mango [9].

Many studies on peaches drying are focused on drying characteristics [112, 113], while no clear information is available on overall quality evaluation of peach chips and the suitable varieties for processing peach chips. The quality of chips includes organoleptic quality, physical and chemical quality and processing quality, and can be influenced by many complex factors [21]. Principal component analysis (PCA), analytic hierarchy process (AHP) and K-means Cluster (KC) and discriminant analysis (DA) have been used to evaluate the quality changes of fruit and vegetable products, such as peaches [114], wines [115], grapes [116], and pineapples [117]. PCA, as a classic statistical analysis method, can extract the common factor from abundant data, and its objective is to identify a reduced number of principal components (PCs) [118]. PCA has been used to discriminate the quality of peaches [119], carrot chips [120], mango [121], and display the changes in chemical constituents of pomegranate [122]. DA can produce score plots for the analyses [45] and be used to discriminate sweet and bitter almonds [123] and different Longjing tea [124]. PCA and DA are used to identify exotic tropical fruits and classify 113 samples into three groups: the irradiated beverages, the non-irradiated beverages and the irradiated standard solutions [125]. AHP provides quick and automatic qualitative sample differentiation, in particular when quantification or characterization of specific components of matrix is not necessary [126].

Many studies have focused on filtering evaluation indicators and evaluating the quality of fruit and vegetables, but only one mathematical analytical method performed. Evaluation system of the overall quality of yellow peach chips based on precise mathematical anlytical

methods is needed. Therefore, the objectives of this work are to develop mathematical analytical methods to filter the characteristic evaluation indicators and to evaluate the overall quality of yellow peach chips prepared by EPD on the basis of instrumently measured characteristic indicators.

2 MATERIALS AND METHODS

2.1 MATERIALS

Based on the production, the harvesting time and the potential for commercial application, fifteen yellow peach varieties were selected from the main yellow peach producing regions of China, including Pinggu (Beijing City, 117°55’E, 40°12’N), Linyi (Shandong Province, 35°27'E, 118°37’N) and Dalian (Liaoning Province, 122°31'E, 39°10’N) (Table 1). Yellow peaches of similar size and maturity were collected from July to September. Forty yellow peaches were picked for each variety. The degree of ripening was evaluated based on the color (Chinese standard SB/T 10090-1992). Namely, the skin color of the fruit was light yellow and the flesh was slightly hard.

Table 1 Germplasm names and collection time and place of 15 yellow peaches samples

No.	Name	Collection Time	Collection Place
1	*Yellow fresh peach*	7/22/2012	Pinggu, Beijing
2	*Fulaidelaika*	7/30/2012	Linyi, Shandong
3	*NJC19*	7/30/2012	Linyi, Shandong
4	*Qiulu*	7/30/2012	Dalian, Liaoning
5	*Huangguanwang*	7/30/2012	Linyi, Shandong
6	*Huangjinxiu*	7/30/2012	Linyi, Shandong
7	*Goldbaby*	7/30/2012	Linyi, Shandong
8	*Guantao 5*	7/30/2012	Pinggu, Beijing

9	*Goldbaby 5*	8/10/2012	Pinggu, Beijing
10	*Juhuang*	8/24/2012	Pinggu, Beijing
11	*Jinlu*	8/24/2012	Dalian, Liaoning
12	*Sengelin*	8/24/2012	Pinggu, Beijing
13	*Goldbaby 6*	8/24/2012	Pinggu, Beijing
14	*Goldbaby 7*	8/24/2012	Pinggu, Beijing
15	*Goldbaby 8*	9/5/2012	Pinggu, Beijing

2.2 SAMPLE PREPARATION AND PROCESSING

Yellow peaches were cut into 9 mm thick slices by a Laboratory Slicer (model FA-200, Nanhai Defeng electrothermal equipment Co., Ltd., Guangdong China) after removing skin and stone. The sliced peaches were placed in a deep-freezer at -80 ^{o}C for 12 h. The sliced fruits were steeped into malt syrup (25%, v/v) for 4 h after thawing at room temperature. Tissue paper was gently used to remove superficial excess water and syrup from outer surface of sample, which might influence the drying characteristics of peach slices. Hot-air drying was used to pre-dry the samples (3.5 h) to get lower water content.

After the above pretreatment procedure, chips were produced by using the experimental EPD equipment system developed by Tianjin Qin-de New Material Scientific Development Co. Ltd. (Tianjing, China). Pre-treated peach slices were arranged in the puffing tank at 85 ^{O}C for 20 min, then the snuffle valves of the EPD equipment were opened to obtain a rapid decompression to approximately 4 KPa (absolute pressure) in the puffing chamber. After that, samples were dried under vacuum conditions at 60 ^{O}C until reaching final moisture content (< 0.075 kg/kg d.b.) [127]. All the samples were stored in a dryer until used.

2.3 COLOR

The surface color of yellow peach chips was analyzed by a colorimeter (D25LT, Hunter Lab, Virginia, USA). The colorimeter was calibrated against a standard white plate before each actual color measurement. Hunter three-color parameter scale was used: L (lightness), a (redness/greenness), and b (yellowness/blueness) [128].

2.4 REHYDRATION RATIO (RR)

Dehydrated samples (3 g) were placed in a baker with distilled water (150 mL) at room temperature for 120 mins. Then the samples were removed and allowed to drain over a mesh for 60 s in order to eliminate the superficial water before weighing [129]. The degree of rehydration ration (RR) was expressed by the formula:

$$RR = m_2 / m_1 \quad (1)$$

where, m_1, m_2 referred to weight of samples (g) before and after rehydration, respectively.

2.5 HARDNESS AND CRISPNESS

Texture profile analysis was used to evaluate the hardness and crispness of yellow peach chips (diameter was 2 cm) by using a texture analyzer (TA. XT 2i/50, Stable Micro Systems Ltd., Godalming, UK) fitted with a spherical probe (p/2.5 S). The maximum compression force and the number of peaks in the force-deformation curve of each sample were used as an indicator of hardness and crispness of sample, respectively.

The analysis parameters were mode: measure force compression; option: return to start; pre-test speed: 1.0 mm/s; test speed: 1.0 mm/s; post-test speed: 2.0 mm/s; distance: 50.0%; trigger force: 100 g; trigger type: button; data acquisition rate: 500 pps.

2.6 MOISTURE CONTENT

The moisture content of peach chips was determined by weight loss after drying 5 g samples in a forced air oven at a given temperature 105 ℃ (AACC, 1986) for 24 h.

2.7 EXPANSION RATIO

Expansion ratio (ER) was expressed as the changes of volume of fresh samples and chips, which was measured using a VolScan Profilter (Stable MicroSystem, England).

$$ER = V_t / V_0 \quad (2)$$

where, V_t and V_0 referred to volume of the sample after and before puffing (cm^3), respectively.

2.8 SOLUBLE SOLID CONTENT (SSC)

Soluble solid content (SSC) was determined by using a Digital Refractometer at 20 ℃ (dBX-55, Atago, Japan).

2.9 Other evaluation indicators

Reducing sugar content, titratable acidity content, ascorbic acid content, crude fiber content, crude protein content and crude fat content were determined according to standard AOAC Official Method 945.66 (1945), AOAC Official Method 942.15 (1942), AOAC Official Method 985.26 (1985), AOAC Official Method 992.16 (1992), AOAC Official Method 978.04 (1978), AOAC Official Method 2003.06(2006), respectively.

2.10 Statistical analysis

All experiments were performed in triplicate and the results were reported as mean values with standard deviations. All data were processed and analyzed using SPSS 21.0 (IBM, Chicago, USA). The method of PCA was applied to integrate the 17 individual quality evaluation indicators of yellow peach chips. The weight and the score of quality characteristic evaluation indicators were obtained by AHP. KC and DA were used to establish the scientific evaluation models.

3 Results and discussion

3.1 Quality evaluation indicators of yellow peach chips

Statistical characteristics of seventeen evaluation indicators of yellow peach chips are shown in Table 2. The coefficient of variation (CV) varied from 3.58 % to 852.89 % between dried products from 15 varieties yellow peaches, signifying that the degree of variation of the indicators was different, which closely related to fruit color, firmness, sugar and acid [130]. The different genotypic variations and texture of fresh fruit might also result in the quality variability among yellow peach chips. The biggest CV was observed in ER (825.89 %). While, the smallest CV was obtained in soluble solid content (3.58 %), indicating no significant difference between the fifteen varieties, and it could be excluded for further data analysis.

Table 2 Variation of 17 indicators for yellow peach chips

Indicators	Average	SD	Minimum	Maximum	CV (%)	Significance
OR (g/g)	0.16	0.03	0.12	0.24	16.90	**
ER (cm^3/cm^3)	1.58	13.45	-33.73	22.33	852.89	**
RR (g/g)	192.02	15.84	154.41	224.92	8.25	**
L value	35.8	4.6	28.8	45.2	12.8	**
a value	10.5	1.4	8.3	14.2	13.0	**

b value	15.8	3.1	10.6	21.6	19.5	**
Water content (g/g)	2.99	0.66	2.01	4.56	22.25	**
Vitamin C content (mg/100g)	11.49	4.82	5.76	22.92	41.96	**
Titratable acid conten t(g/100g)	5.30	1.36	3.05	7.54	25.61	**
Crude protein content (g/100g)	2.61	1.67	0.75	5.08	63.96	**
Crude fat (g/100g)	0.68	0.42	0.32	2	61.11	**
Crude fiber content (g/100g)	3.25	0.65	2.20	4.76	19.90	**
Reducing sugar content (g/100g)	4.42	3.87	1.36	9.075	87.46	**
Hardness (g)	6153.2	8684.1	190.1	28986.9	141.1	**
Crispness (s)	16.3	6.4	1.6	27.0	39.6	**
Sugar-acid (%)	0.77	0.59	0.22	1.69	77.34	**
Soluble solid content (g/g)	93.81	3.36	85.34	99.00	3.58	--

Note: ** $p<0.01$; SD, standard deviations; CV, coefficient of variation; OR, out-put ratio; ER, explosion ratio; RR, rehydration ratio.

3.2 Principal component analysis (PCA)

PCA is a technique to both reduce the number of variables and give prominence to the relationship between the elements [131]. The number of the characteristic quality evaluation indicators can be determined based on Eigenvalues (Kaiser's rule). The Eigenvalues of the first five components were greater than 1, and explained 85.84% of the total variance (Table 3). The PC1 explained 35.376 % of total variability, and it was negatively correlated with crude fat content and positively with reducing sugar content. PC2 (explained 20.690 %), it was positively correlated with RR, and negatively connected with OR. PC3 (explained 12.636 %) was only positively correlated with a value. PC4 (explained 10.458 %),

was positively correlated with L value and b value. PC5 (explained 6.681 %) was only positively correlated with water content.

	Principal Components (PCs)				
	1	2	3	4	5
Crude fat	**-0.856**	0.267	0.015	0.025	0.108
Reducing sugar	**0.855**	0.387	-0.209	-0.139	0.020
Sugar-acid ratio	0.814	0.414	-0.223	-0.125	0.210
Vitamin C	0.648	0.257	0.204	-0.550	-0.199
RR	0.311	**0.910**	0.044	-0.203	-0.012
OR	-0.038	**-0.872**	-0.154	0.219	0.198
Total dietary fiber	0.074	0.732	-0.266	-0.073	-0.016
Hardness	-0.061	-0.669	-0.290	-0.221	0.239
a value	-0.177	-0.004	**0.928**	0.147	-0.008
Crude protein	0.618	0.263	0.697	-0.139	-0.008
ER	-0.041	0.083	0.642	-0.302	-0.515
Cispness	-0.394	-0.131	0.580	0.044	-0.429
L value	-0.047	-0.089	-0.112	**0.963**	0.170
b value	-0.223	-0.093	0.188	**0.937**	0.045

Water content	0.020	-0.200	-0.211	0.138	**0.903**
Reducing acid	0.564	0.254	-0.173	-0.184	-0.632
Eigenvalue	5.660	3.310	2.022	1.673	1.069
ercent of variance (%)	35.376	20.690	12.636	10.458	6.681

Table 3 Factor analyses of 16 indicators of yellow peach chips

Note: RR, OR and ER represent rehydration ratio, out-put ratio and explosion ratio, respectively.

PCA can seek the most economical basis to represent each individual term, so each term can be well described by a linear combination of only a few basic formation. Both crude fat content and reducing sugar content with large weight values were the main indicators for PC1. However, yellow peach chips produced by EPD were fried-free, and low fat content ranged from 0.75 % to 5.08 %. Reducing sugar content was related to both browning degree and the taste of yellow peach chips, which could reflect the sweetness. As a substrate of Maillard reaction, reducing sugar content is responsible for the heat-induced browning reaction during the drying processing. Keenan et al. [132] reported that reducing sugar content was associated with high browning tendency during drying processing, since Maillard reaction was occurred at higher temperatures. Therefore, reducing sugar content can be selected as the representative indicators.

Both RR and OR with the large values of weight were the main indicators for PC2. RR was considered as an indirect measurement of the damage to the material caused by drying and treatment preceding dehydration [133]. It is an indicator not only for evaluating process ability, but also for evaluating the texture of yellow peach chips. Compared with OR, RR can better characterize the physical and chemical changes of yellow peach chips as influenced by processing conditions [134]. Thus, RR was used as the representative indicator to evaluate the structure of yellow peach chips, and selected as the characteristic indicator of PC2.

For PC3 and PC4, both a value (0.928) and L value (0.963) got the largest weight, and could be defined as color indicators. Color was an extremely significant factor for the quality evaluation of dehydrated fruit [134]. L value and a value were closely related to browning indicators that were induced by enzymatic or non-enzymatic browning. As reported by Ávila [135], with the increasing heating temperature and time, peach puree became darker corresponding to the decrease of a value. Therefore, a value and L value could be selected as characteristic indicators for PC3 and PC4, respectively.

Water content is an important indicator to evaluate the safety of chips products. Water content of the product can affect microbial spoilage and quality deterioration due to undesirable biochemical reactions [136]. Therefore, water content with the largest weight (0.903) can be regarded as the characteristic indicator for PC5.

In summary, five characteristic indicators of yellow peach chips are obtained by PCA. They are reducing sugar content, RR, a value, L value and water content.

3.3 Analytic Hierarchy Process (AHP)

In order to eliminate the influence caused by the different dimensions and order of magnitudes of the evaluation indicators, the original data of evaluation indicators are standardized. According to the procedure of AHP, three layers (Fig. 4) of the hierarchy model was constructed based on the membership level among the chips. The first layer was objective (O), meaning the comprehensive orders; the second layer was criterion (C), meaning four types of evaluation indicators, marked C= (c_1,c_2,c_3,c_4,) = (tasty quality, texture quality, safety quality, color quality); the third layer was project (P), meaning the five characteristic indicators, marked P=(P1, P2, P3,P4, P5) = (reducing sugar content, RR, water content, a value, L value). Secondly, an additive scale ranging from 1 to 9 is used to establish the quality evaluation judgment matrix of yellow peach chips (Table 4). When the ratio of consistency (0.038) is lower than 0.10, the quality evaluation judgment can be considered as reasonable and logical. The weights of characteristic evaluation indicators of yellow peach chips were 0.0429, 0.1140, 0.4816, 0.1807, and 0.1807, respectively.

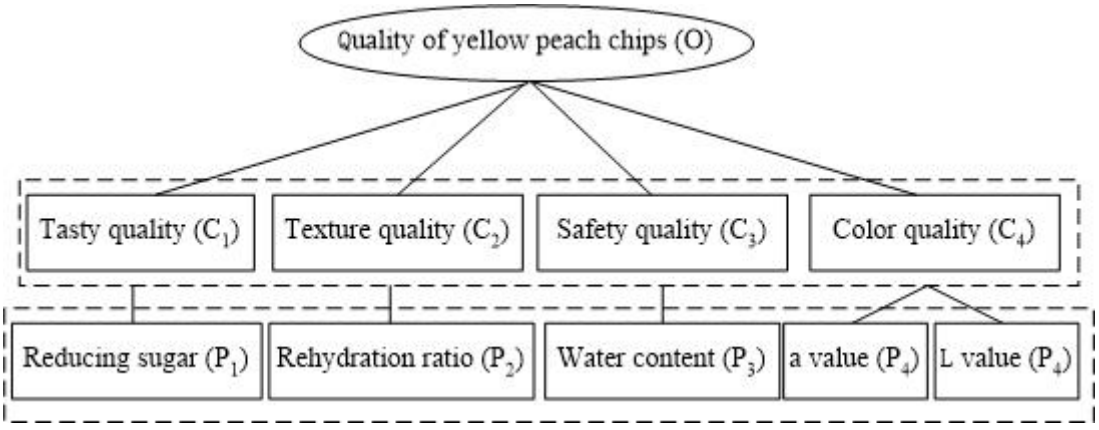


Figure 4 Hierarchical structure of characteristic evaluation indicators analyzed by AHP for yellow peach chips prepared by EPD

Note: O, objective; C, criterion; P, project. C_1 - C_4 represented tasty quality, texture quality, safety quality and color quality, respectively; P_1 - P_5 represented reducing sugar, rehydration ratio, water content, a value and L value, respectively.

Table 4 Quality evaluation judgment matrix of yellow peach chips

	P_1	P_2	P_3	P_4	P_5	weight
P_1	1	1/3	1/7	1/5	1/5	0.0429
P_2	3	1	1/3	1/2	1/2	0.1140
P_3	7	3	1	4	4	0.4816
P_4	5	2	1/4	1	1	0.1807
P_5	5	2	1/4	1	1	0.1807

Note: P1-P5 represent reducing sugar content, rehydration ratio, water content, a value and L value, respectively.

The information of the scores of overall quality of yellow peach chips were presented in Table 5, “sengelin” with best overall quality had the the highest score and “Goldbaby” with the worst overall quality had the lowest score.

Table 5 the quality evaluation indicators standardized data and scores of overall quality of yellow peach chips

Varieties	Reducing sugar	RR	Water content	a value	L value	score	Ranking
Weight	0.0429	0.1140	0.4816	0.1807	0.1807		
Senggelin	1.1818	0.6391	1.5626	-1.6205	2.0569	11.1037	1
Huangjinxiu	-0.7850	-2.3738	2.3648	-0.4383	0.2961	2.5698	2
Delaifulaika	-0.7910	-0.2609	0.2440	2.7233	1.3195	-2.4193	3
GNC19	-0.7917	0.6147	0.9610	0.6245	0.2888	0.5407	4
Goldbaby 8	1.1921	2.0762	0.1738	-0.0093	-1.0002	0.9650	5

Qiulu	-0.7904	-0.8150	-0.0267	-0.1360	0.3537	-0.5488	6
Jinlu	1.2046	0.5500	0.1036	-0.4553	-1.1293	0.3520	7
Guangtao 5	-0.7894	0.0394	-0.7287	0.6489	0.3252	-2.4289	8
Goldbaby 7	1.1580	0.7375	-0.6936	0.5319	-0.6872	-1.5195	9
Yellow fresh peach	-0.7915	-0.8713	-0.1922	0.0029	-0.0658	-1.9325	10
Juhuang	1.1774	-0.0439	-0.6635	-1.0355	1.0218	4.1065	11
Goldbaby 6	1.1852	0.4708	-0.4228	-0.0385	-1.5188	-2.0209	12
Huanguanwang	-0.7860	0.0110	-0.1721	-1.1306	-1.1599	-1.7446	13
Goldbaby 5	-0.7842	0.0634	-1.0395	0.0273	-0.3444	-3.2633	14
Goldbaby	-0.7897	-0.8371	-1.4707	0.3052	0.2435	-3.7600	15

Note: RR represents rehydration ratio.

In this study, AHP was applied to obtain the weights of five characteristic evaluation indicators and achieve the scores for overall quality of yellow peach chips. However, the additive scale ranging from 1-9 was not the only scale used in the evaluation method. One of the most widely cited alternative scales was the geometric scale, which used the range (e^{0y} to e^{8y}) for the same semantic descriptions as the additive scale ranging from 1-9 [137]. Hence, more additive scales could be applied in AHP, but a ratio characterization should be used for the purpose.

Quality evaluation of yellow peach chips was a complex multi-dimensional process, involving multiple criteria and factors. Multi-criteria methods could be served as useful aids for carrying out the quality evaluation. AHP has become a method used in solving various multi-criteria problems [138], and also has been applied to numerous practical problems in the last decades.

3.4 K-MEANS CLUSTER (KC) AND DISCRIMINATE ANALYSIS (DA)

Based on the methods of KC and DA, the overall quality of yellow peach chips from 1 varieties allowed us to divide the samples of fruit into three grades, namely, excellent, medium and bad. The discriminant functions were established to characterize the overall quality of yellow peach chips from different varieties:

$$F_1 = -31.972 + 1.386 \times x_1 + 4.458 \times x_2 + 32.759 \times x_3 + 14.663 \times x_4 + 17.104 \times x_5$$
$$F_2 = -14.936 - 1.549 \times x_1 - 2.904 \times x_2 - 21.752 \times x_3 - 9.978 \times x_4 - 11.563 \times x_5$$
$$F_3 = -2.273 + 0.093 \times x_1 - 0.888 \times x_2 - 6.290 \times x_3 - 2.629 \times x_4 - 3.166 \times x_5$$

where, x_1 - x_5 were reducing sugar content, rehydration ratio, water content, a value and L value, respectively.

The diversity in overall quality of yellow peach chips within each group and their relationship were shown in Fig. 5. The group scatter plot represented the highest possible correlation between linear combination of overall quality of yellow peach chips. Here, the score of the first characteristic vector (F1) and second characteristic vector (F2) for all the yellow peach chips are represented in x-axis and y-axis, respectively. In the group scatter plot graph (Fig. 5), a wide discrimination between the overall qualities of yellow peach chips could be separated by the characteristic evaluation indicators weight (Table 4). The discriminant values of the overall quality of yellow peach chips confirmed the influence of the characteristic evaluation indicators as the differentiating elements.

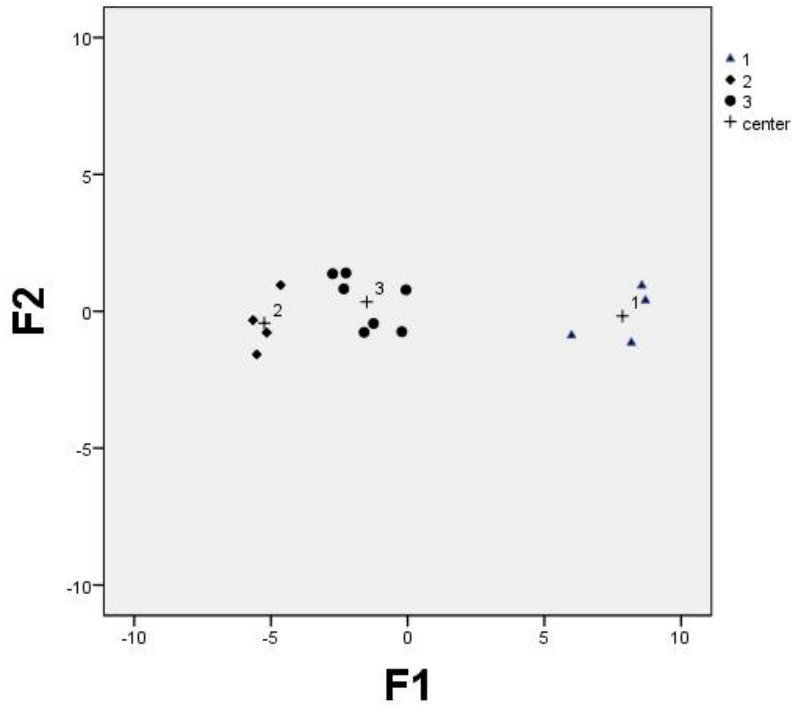


Figure 5 Group scatter plot of yellow peach chips with different quality on canonical discrimination function by discriminant function

NOTE: 1 presented the excellent varieties, including Senggelin, Huangjinxiu and Delaifulaika; 2 represented bad varieties, including Goldbaby 6, Huangguanwang, Goldbaby 5 and Goldbaby; 3 represnted medium varieties, including GNC 19, Goldbaby 8, Qiulu, Jinlu, Guangtao 5, Goldbaby 7, yellow fresh peach and Juhuang.

F1 and F2 represented the score of the first characteristic vector and second characteristic vector of yellow peach chips, respectively.

Montevecchi [139] proved that the quality of peach could be affected by the physio-chemical and sensory indicators, including color, sugars, weight and so on. Thus, PCA can be used to simplify the evaluation indicators. The characteristic evaluation indicators and their weights were analyzed to determine whether KC and DA could separate the overall quality of yellow peach chips from different varieties into different levels. The results revealed that the characteristic evaluation indicators were able to forecast the key quality of yellow peach chips. KC and DA could be used as an important analysis tool to evaluate and distinguish the overall quality of fruit or fruit products.

4 CONCLUSIONS

Yellow peaches from 15 different varieties were used to process chips by EPD. As expected, the evaluation indicators except soluble solid content varied significantly between the different varieties. PCA and AHP gave the performance of the five characteristic evaluation indicators from different yellow peach varieties in the processed products. KC and DA was applied to establish the discriminant functions, which were used to separate the overall quality of yellow peach chips into three levels. The excellent varieties for processing chips were Senggelin, Huangjinxiu and Delaifulaika; The medium varieties were GNC 19, Goldbaby 8, Qiulu, Jinlu, Guangtao 5, Goldbaby 7, yellow fresh peach and Juhuang; the bad varieties were Goldbaby 6, Huangguanwang, Goldbaby 5 and Goldbaby. PCA, AHP, KC and DA could be applied to simplify the quality evaluation process and improve its efficiency, with useful application in quality control in the food industry.

CHAPTER 3: QUALITY EVALUATION OF PEACH AND NECTARINE CHIPS PREPARED BY EXPLOSION PUFFING DRYING

This chapter is a modified version of the following research article:

Lyu, J., Liu, X., Bi, J., Zhou, L., Lu, X. (2016). Research on the quality evaluation for peach and nectarine chips by explosion puffing drying. Scientia Agricultura Sinica 49(4): 802-812

The aim of this study was to investigate the variations in the quality of peach and nectarine chips from different varieties and establish scientific models to evaluate the overall quality of peach and nectarine chips. Forty-nine varieties of peach and nectarine fruit grown in the north of China were selected as test materials. Seventeen quality evaluation indicators were measured, including organoleptic quality indicators, physical and chemical characteristic indicators and processing quality indicators. A variable coefficient method was used to investigate the differences in quality evaluation indicators for different peach and nectarine varieties. A factor analysis was applied to the evaluation indicators. The weights and the levels of the characteristic indicators were calculated by an analytic hierarchy process (AHP) and a range analysis method, respectively. The levels in varieties effectively were classified by discriminant functions which were obtained by K-means cluster and discriminant analysis. Results showed that the 17 quality evaluation indicators varied widely with a coefficient of variation ranging from 0.70% - 344.02%. The five characteristic indicators explained 74.626% of the total variances. Reducing sugar content, rehydration ratio, L value, crude protein content and expansion ratio were selected as the characteristic indicators. Based on the AHP, the weights of the characteristic indicators were 0.0824, 0.1724, 0.2732, 0.0048 and 0.4240, respectively. The scoring standard of the characteristic indicators were also established. Discriminant functions of different grades were established for the test samples, which had satisfactory recognition accuracy up to 100%. Only one test sample was discriminated inaccurately. Rupan 19, Delaifulaika, and Dajiubao were the best varieties used to produce peach and nectarine chips. Ruipan 20, Sengelin and Huangjinxiu were the worst varieties used to process peach chips. Discriminant functions were shown to be effective in discriminating the quality of peach and nectarine chips.

Key words: peach chips, quality evaluation, characteristic indicators, factor analysis, analytics hierarchy process, discriminant analysis

1 INTRODUCTION

Peaches and nectarines are the fourth most important fruit group in China, after oranges, apples and pears. China has been the leading grower and producer of peaches and nectarines in the world since 1993. In 2014, the production and harvested areas of peaches and nectarines were 12,452,377 t and 728,354 ha, respectively (FAO, 2014). However, the export value of peach and nectarine of China was only 44.439 (× 1000 USD), which only accounted for 1.75% of the world export value. The export price of peaches and nectarines from China was only 1/3 of the average global export price. Peaches and nectarines have a short shelf-life potential due to fast softening and overall ripening [140]. Therefore, drying technologies to produce peach and nectarine products provide potentially valuable alternatives to fresh products.

In this paper, we sought to evaluate qualitative differences in peach and nectarine chips produced from different varieties. Additionally, we aimed to propose discriminant functions to evaluate the overall quality of peach and nectarine chips. Drying is one of the most important technologies to preserve and extend the shelf-life of peaches and nectarines. Many studies on peach drying technology have been reported. The effect of various parameters of explosion puffing drying (EPD) process on expansion ratio (ER) and color quality of peach and nectarine chips were reported by He et al. [141]. Wang et al. [142] conducted a series of microwave-far-infrared drying tests on yellow peaches and calculated the best drying parameter. Chang et al. [143] selected color, crispness and water content as the quality evaluation indicators of peach chips dehydrated by microwave-vacuum drying and studied the optimal combination parameters. Li et al. [144] selected water content and fat content as the quality evaluation indicators of peach chips dehydrated by combined vacuum-frying and hot-air drying and modified the vacuum-frying temperature and hot-air drying temperature to improve the finished products. Some studies [113, 145] focused on the influence of high-pressure blanching and drying air temperature on the drying characteristics of peaches and nectarines. Germer et al. [146] studied the influence of process variables on the osmotic dehydration (OD) and sensory tests of sliced dehydrated peaches. Color, texture and flavor were selected as the quality evaluation indicators in their study. Therefore, different quality evaluation indicators need to be used due to the differences between the different drying technologies.

There is a lack of systematic research on overall quality indicators of peach and nectarine chips. Recently, precise mathematical analysis has been applied to evaluate the overall quality of fruits and fruit products. Zhang et al. [147] used principal component analysis (PCA) and systematic cluster analysis to select characteristic indicators which could be used as a comprehensive system for evaluating peach and nectarine quality. Versini et al. [118] applied PCA to reveal the variables of specific aroma fractions, and could distinguish

between the varieties. Liu et al. [148] used an analytic hierachy process (AHP) to select four varieties of Jinhua pear from 12 varieties based on multifactor evaluation of seven quality evaluation indicators. PCA and DA were successfully used by Dourtoglou et al. [149] to discriminate wines produced from different varieties when no other information was available.

Many studied have focused on evaluating the quality of peach and nectarine chips, but the included few qualitative indicators. Evaluation system of the overall quality of peach and nectarine chips based on precise mathematical analysis is needed. The objective of this study was to characterize the peach and nectarine chips from different varieties grown in Northern China, and to discriminate the overall quality of peach and nectarine chips on the basis of instrumentally measured characteristics.

2 Materials and Methods

2.1 Materials

Based on the production, the harvesting time and the potential for commercial application, forty-nine peach and nectarine varieties were selected from the main peach producing regions of China, including Dalian (Liaoning Province), Linyi (Shandong Province) and Yangling (Shanxi Province) (Table 6). Peaches and nectarines of similar size and maturity (Chinese standard SB/T 10090-1992) were collected. Forty peaches and nectarines were picked for each variety. The degree of ripening was evaluated based on the color. Briefly, the skin color of the fruit varied from light green to light yellow, depending on the varieties. The flesh was slightly hard and exhibited yellow or red ground color, also depending on the variety.

Table 6 Germplasm names and collection time and place of peach and nectarine samples

No	Name	Collection time	Collection place	No	Name	Collection time	Collection place
1	Hongburuan	7/22/2012	Dalian, Liaoning	26	Qiulu	8/24/2012	Pinggu, Beijing
2	Zaoyu	7/22/2012	Dalian, Liaoning	27	Huangguanwang	8/24/2012	Pinggu, Beijing
3	Italy 5	7/22/2012	Dalian, Liaoning	28	Huangjinxiu	8/24/2012	Pinggu, Beijing
4	Ruiguang 51	7/22/2012	Dalian, Liaoning	29	Ruiguang 39	8/24/2012	Pinggu, Beijing

5	Ruiguang 28	7/22/2012	Dalian, Liaoning	30	Huayu	9/05/2012	Linyi, Shandong
6	Ruiguang 29	7/22/2012	Dalian, Liaoning	31	Wan 9	9/05/2012	Linyi, Shandong
7	Flat peach 19	7/22/2012	Dalian, Liaoning	32	Yanfeng 1	9/05/2012	Linyi, Shandong
8	Huge flat peach	7/22/2012	Dalian, Liaoning	33	Goldbaby	9/05/2012	Linyi, Shandong
9	Dajiubao	8/10/2012	Pinggu, Beijing	34	Guantao 5	9/05/2012	Linyi, Shandong
10	Jingyu	8/10/2012	Pinggu, Beijing	35	Goldbaby 5	9/05/2012	Linyi, Shandong
11	Huangyoutao	8/10/2012	Pinggu, Beijing	36	Hanlumi	9/05/2012	Pinggu, Beijing
12	Yellow peach	8/10/2012	Pinggu, Beijing	37	Cuiyu	9/10/2012	Pinggu, Beijing
13	Ruiguang 18	8/10/2012	Pinggu, Beijing	38	Dadongtao	9/10/2012	Pinggu, Beijing
14	Ruiguang 20	8/10/2012	Pinggu, Beijing	39	Wan 24	9/10/2012	Pinggu, Beijing
15	Ruiguang 27	8/10/2012	Pinggu, Beijing	40	Bayuecui	9/10/2012	Pinggu, Beijing
16	Flat peach 4	8/10/2012	Pinggu, Beijing	41	Juhuang	9/10/2012	Pinggu, Beijing
17	20 Ruipan 20	8/10/2012	Pinggu, Beijing	42	Goldbaby 6	9/10/2012	Pinggu, Beijing
18	21 Ruipan 21	8/10/2012	Pinggu, Beijing	43	Goldbaby 7	9/10/2012	Pinggu, Beijing
19	Yanhong	8/24/2012	Pinggu, Beijing	44	Sengelin	9/10/2012	Pinggu, Beijing
20	Jingmi	8/24/2012	Pinggu, Beijing	45	Jinlu	9/10/2012	Pinggu, Beijing
21	Guibao	8/24/2012	Pinggu, Beijing	46	2000-6-9 east	9/17/2012	Yangling,

							Shanxi
22	Guantao 14	8/24/2012	Pinggu, Beijing	47	Yanhong	9/17/2012	Yangling, Shanxi
23	Jingyan	8/24/2012	Pinggu, Beijing	48	Goldbaby 8	9/17/2012	Yangling, Shanxi
24	Delaifulaika	8/24/2012	Pinggu, Beijing	49	Flat peach 3	9/17/2012	Yangling, Shanxi
25	NJC19	8/24/2012	Pinggu, Beijing				

2.2 METHODS

2.2.1 SAMPLE PREPARATION AND PROCESSING

Peaches and nectarines were cut into 9mm thick slices by a laboratory Slices after removing skin and stone. The sliced peach and nectarine were placed in a freezer at -80 ^{o}C for 12 h. After thawing at room temperature, the sliced samples were steeped into malt syrup (25 %, v/v) for 4 h. Hot air drying was used to pre-dry the samples (3.5 h) to get lower water content (0.5 kg/kg dry base, d.b.). After the above pretreatment procedure, chips were produced by using the experimental explosion puffing drying (EPD) equipment system (QDPH 1021, Tianjin Qin-de New Material Scientific Development Co. Ltd., Tianjin, China). Pre-treated samples were arranged in the puffing tank at 85 ^{o}C for 20 min, then the snuffle valves of the EPD equipment were opened to obtain a rapid decompression to approximately 4 KPa (absolute pressure) in the puffing chamber. After that, the samples were dried under continuous vacuum at 60 ^{o}C until reaching final moisture content (< 0.075 kg/kg d.b.). All the samples were stored in a dryer until used.

2.2.2 INDICATOR DETECTION

organoleptic quality indicators:

The surface color of peach and nectarine chips was analyzed by a colorimeter (D25L, Hunterlab, Virginia, USA). Hunter three-color parameter scale was used: L (lightness), a (green-blue hue), b (blue-yellow) values were measured.

Texture profile analysis was used to evaluate the hardness and crispness of the fruit. The diameter of the peach and nectarine chips was about 2 cm. The texture analyzer (TA. XT 2i/50, Stable Micro Systems Ltd., Godalming, UK) has a spherical probe (p/2.5s). The maximum compression force and the number of peaks in the force-deformation curve of

each sample were used as an indicators of hardness and crispness, respectively. The parameters were mode: measure force compression; option: return to start; pre test speed: 1.0 mm/s; test speed: 1.0 mm/s; post test speed: 2.0 mm/s; distance: 50%; trigger force: 100 g; trigger type: button; data acquisition rate: 500 pps.

ER was expressed as the changes of volume of fresh sample and chips [141], which was measured using a VolScan Profiler (Stable MicroSystem, UK). ER was calculated using the following equation:

$$\mathrm{ER} = V_t/\mathrm{V}_0 \quad (3)$$

where, V_t and V_0 referred to volume of the sample after and before puffing (cm^3), respectively.

Reducing sugar content and Titratable Acidity content were determined according to the AOAC Official Method 923.09 and 942.15, respectively.

Physical and chemical quality indicators:

Water content was determined based on China standard GB 5009.3 (2001). Briefly, samples were arranged in the hot-air drying equipment at a given temperature (115 ^{o}C) until the constant weight was reached.

Soluble solids content was determined by using a refractometer according to Chinese standard GB/T 12295 (1990). A titration with dichlorophenolindophenol (GB/T 6195, 1986) was used to measuring the content of Vitamin C.

Soxhlet extractor method (GB/T 14772, 2008), Gravimetrical method (GB/T, 5009.5, 2010) and Kjeldahl method (GB/T 6195, 1986) were applied to determine the content of crude fat, crude fiber and crude protein, respectively.

Processing quality indicators:

Output ratio (OR) and rehydration ratio (RR) were determined using the method described by Irfan et al. [150] and Germer et al. [146], respectively, with some modification, respectively. The equations for calculating OR and RR were as follows:

$$\mathrm{OR} = m_2/m_1 \quad (3)$$

where m_1 and m_2 referred to weight of samples (g) before and after EPD, respectively.

$$\mathrm{RR} = M_2/M_1 \quad (4)$$

where, M_1 and M_2 referred to weight of samples (g) before and after rehydration, respectively.

2.2.3 EQUIPMENTS

Deep-freezer (MDF-U50V, Sanyang Co., Ltd, Beijing, China), laboratory slicer (model FA-200, Nanhai Defeng Electro Thermal equipment Co., Ltd., Guangdong, China), Volscan Profileer (VSP 3000045, Stable MicrSystem, Godalming, England), colorimeter (D25LT, Hunter Lab, Osaka, Japan), texture analyzer (TA.XT 2i/50, Stable Micro Systems Ltd., Godalming, UK), drying oven (DHG-9030, Jing Hong Laboratory Instrument Co., Ltd., Shanghai, China), spectrophotometer (UV-1800, Shimadzu, Kyoto, Japan), refractometer (MASTER-a, Atago Co. Ltd., Tokyo, Japan), EPD equipment (QDPH 1021, Tianjin Qin-de New Material Scientific Development Co. Ltd., Tianjin, China).

2.3 STATISTICAL ANALYSES

All experiments were performed in triplicate. All data were processed and analyzed using SPSS 21.0 (IBM, Chicago, IL, USA). The method of factor analysis (FA) was applied to integrate the 17 individual quality evaluation indicators of peach and nectarine chips. The weight and the score of quality characteristic evaluation indicators were obtained by AHP and frequency analysis. K-means cluster (KC) and discriminate analysis (DA) were used to establish the scientific evaluation models.

3 RESULTS

3.1 QUALITY EVALUATION INDICATORS OF PEACH AND NECTARINE CHIPS

Statistical characteristics of 17 evaluation indicators of peach and nectarine chips were shown in Table 7. The coefficient of variation (CV) varied from 0.70%-344.02% between dried products from 49 varieties, signifying that the degree of variation of the different indicators was different. The CV of soluble solid content was the lowest (< 10%) due to the similar degree of maturity between the specimens of different varieties, indicating no significant differences among the 49 varieties. Therefore, soluble solid content could be excluded from further data analysis. The biggest CV was observed in ER (344.02%), which closely relates to fruit characteristics including genotype, polysaccharide content, texture, and so on. The mean value was close to the median value for each indicator except the indicator of hardness, indicating few outliers [151]. These results show that the quality of peach and nectarine chips dehydrated by EPD differed significantly between the 49 varieties.

Table 7 Variation of 17 indicators of peach and nectarine chips

No.	Indicator	Range	Mean	Median	Standard deviation	*CV* (%)
1	OR (%)	0.12—0.32	0.20	0.19	0.05	23.45
2	ER (%)	-37.51—23.08	5.97	5.90	13.83	344.02
3	RR (%)	144.35—232.96	181.95	178.21	20.54	11.29
4	L value	28.8—51.3	37.8	36.9	6.2	16.4
5	a value	5.2—14.2	9.3	9.4	1.7	18.5
6	b value	10.6—21.6	15.2	14.8	2.7	17.8
7	Moisture (%)	0.82—5.66	2.54	2.50	0.88	34.79
8	Vitamin C (mg/100g)	5.74—27.02	12.26	11.81	4.19	40.08
9	Titratable acid (%)	0.16—0.51	0.29	0.26	1.19	27.96
10	Crude protein (mg/100g)	0.75—5.08	2.12	1.92	1.18	55.57
11	Crude fat (mg/100g)	0.18—2.00	0.49	0.39	0.30	60.56
12	Crude fiber (mg/100g)	2.0—4.76	3.36	3.36	0.62	18.35
13	Reducing sugar (%)	0.92—9.08	5.47	6.02	3.52	64.38
14	Hardness (g)	190.1—29877.7	8394.2	4747.6	8664.4	103.2
15	Crispness	1.6—31.3	13.4	12.8	7.3	54.9
16	Sugar-acid ratio	3.27—40.16	20.02	21.63	13.57	67.77
17	Soluble solid (%)	73.98—98.07	89.99	92.37	6.39	0.70

Note: CV: coefficient of variation; OR, ER and RR represented out-put ratio, explosion ratio and rehydration ratio, respectively.

3.2 CHARACTERISTIC QUALITY EVALUATION INDICATORS

The total variance explained in the FA was shown in Table 8. The number of the characteristic quality evaluation indicators could be determined based on the Eigenvalues and screen plot of FA (Figure 6). The Eigenvalues of first five components were greater than 1, and explained 74.626% of the total variance (Table 8). From the sixth component, the Eigenvalues trended to zero. Therefore, the dimensionality was reduced to five principal components (PCs).

Table 8 Total Variance Explained by Factor Analysis

Factor number	Eigenvalue (λ)	Cumulative variance contribution (%)	Factor number	Eigenvalue (λ)	Cumulative variance contribution (%)
1	4.037	25.233	9	0.491	91.147
2	3.361	46.238	10	0.386	93.561
3	2.302	60.626	11	0.319	95.556
4	1.207	68.169	12	0.293	97.385
5	1.033	74.626	13	0.206	98.674
6	0.769	79.434	14	0.149	99.606
7	0.736	84.032	15	0.051	99.926
8	0.648	88.080	16	0.012	100.00

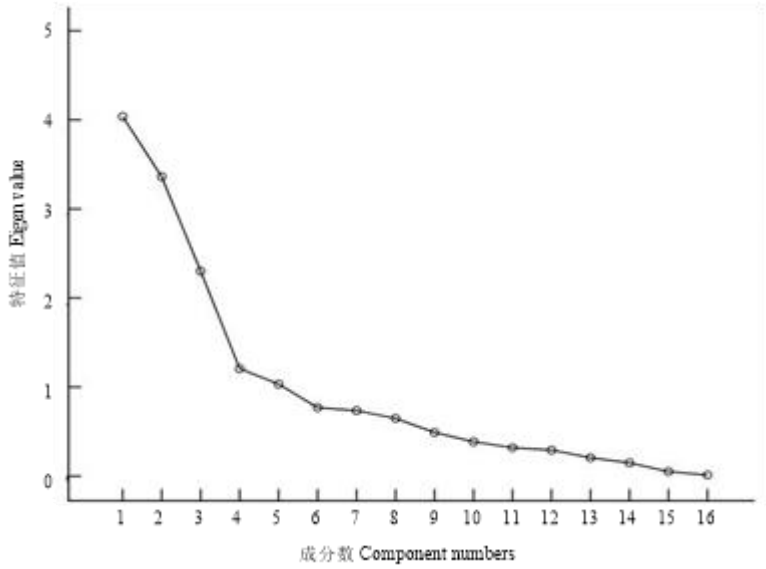


Figure 6 Screen plot of factor analysis

To clarify the details of each PC, all the evaluation indicator values were further analyzed by varimax rotation. Varimax rotation is a modification of coordinates used in FA that maximizes the sum of the variances of the squared loadings. It seeks the most economical basis to represent each individual term, so that each term can be well described by a linear combination of only a few basic functions. From Table 9, reducing sugar content and sugar-acid ratio were positively correlated with PC1, indicating the taste quality of peach and nectarine chips. The weight of reducing sugar content (0.870, in absolute value) was higher than that of sugar-acid (0.853). In addition, a significant correlation of 0.904 between reducing sugar content and sugar-acid was founded, therefore, reducing sugar content can be selected as the representative indicator. PC2 was positively connected with RR, and negatively connected with OR. Rehydration characteristics of dried products are quality parameters that indicated physical and chemical changes occurring during the drying process [152]. Compared with OR, RR could better characterize the physical and chemical changes of peach and nectarine chips as influenced by processing conditions. Thus, rehydration was used as the representative indicator to evaluate the structure of peach and nectarine chips, and selected as the characteristic indicator of PC2. Both L value and b value (the correlation coefficient R=0.77) with large weight values were the main indicators for PC3. L value indicated lightness, and its value ranges from 0 (an ideal black object) to 100 (an ideal white object). Positive b value and negative b value indicated yellow and blue direction, respectively. Based on the pulp color (white or yellow) of peach and nectarine from different varieties, L value could be viewed as a characteristic indicator for PC3. For PC4 and PC5, both crude protein content (0.900) and ER (0.779) got the highest weights. It could be regarded as the characteristic indicator for PC4 and PC5, respectively. In summary, five characteristic

indicators of peach and nectarine chips were obtained by FA. They were reducing sugar content, RR, L value, crude protein content and ER.

Table 9 Rotated component matrix of factor analysis

	PC1	PC2	PC3	PC4	PC5
OR	0.172	**-0.865**	0.029	-0.045	0.111
ER	0.200	0.000	0.189	-0.060	**0.779**
RR	0.090	**0.897**	-0.025	0.048	-0.055
L value	0.072	-0.323	**0.878**	-0.118	0.057
a value	-0.342	0.476	-0.429	0.362	0.159
b value	-0.199	-0.055	**0.871**	0.089	0.131
Moisture content	0.007	0.358	0.529	0.000	-0.588
Vitamin C content	0.335	-0.102	-0.759	0.257	0.057
Titratable acid content	-0.035	0.562	-0.213	0.448	-0.009
Crude protein content	0.100	0.158	-0.070	**0.900**	-0.044
Crude fat content	-0.734	0.205	0.090	-0.181	-0.027
Crude fiber content	0.508	0.401	0.023	-0.316	0.093
Reducing sugar content	**0.870**	-0.021	-0.233	0.189	0.173
Hardness	-0.501	-0.468	0.050	-0.172	-0.399
Crispness	-0.785	-0.210	-0.069	0.261	0.096

Sugar-acid ratio	**0.853**	-0.224	-0.166	-0.017	0.243

Note: Rotation after the eighth interaction convergence. PC1 - PC5 represented the first, second, third, forth fifth principle component, respectively. OR, ER and RR represented out-put ratio, explosion ratio and rehydration ratio, respectively.

3.3 SCORING STANDARD OF THE CHARACTERISTIC INDICATORS

The quality evaluation judgment matrix (Table 5) among the project layer (P) was established based on the method of additive scale ranging from 1 to 9 [148]. When the ratio of consistency (0.0112) is lower than 0.10, the quality evaluation judgment matrix can be considered as reasonable and logical. The weights of characteristic evaluation indicators of peach and nectarine chips were 0.0824, 0.1724, 0.2732, 0.0480 and 0.4240, respectively.

Table 10 Quality evaluation judgment matrix of peach and nectarine chips

	P1	P2	P3	P4	P5	Weight
P1	1	1/3	1/4	3	1/5	0.0824
P2	3	1	1/2	4	1/3	0.1724
P3	4	2	1	5	1/2	0.2732
P4	1/3	1/4	1/5	1	1/5	0.0480
P5	5	3	2	5	1	0.4240

Note: P1 - P5 represented reducing sugar content, rehydration ratio, L value, crude protein content and explosion ratio.

The scores of characteristic indicators for peach and nectarine chips were shown in Table 10. Range analysis was applied to calculate the score of characteristic indicators with different grades. The calculated result of the weight of each characteristic evaluation indicator multiplied by 100 was regarded as the full score for the characteristic evaluation indicator. The total score of the five characteristic evaluation indicators was 100. The highest grade was given the full score. The result of 10% of the full score subtracted from the full score was given to the second grade. The greater the values of L, RR, crude protein content and ER, the better the quality of the peach and nectarine chips obtained. According to Chinese diet characteristics [153], the medium value of reducing sugar was viewed as the highest grade,

and 20% of the full score subtracted from the full score was regarded as the second grade. The remaining scores calculated in the same manner.

3.4 DISCRIMINANT FUNCTIONS

The score for overall quality of peach and nectarine chips was obtained by multiplying the standardized characteristic evaluation indicators by the corresponding weight. Based on the method of KC method, the overall quality of peach and nectarine chips from 49 varieties allowed us to divide the samples of fruits into three grades, namely, excellent, medium and bad. A total of 70% of samples from the three grades were taken as modeling samples, and the remaining samples were used for testing. The discriminant functions were established to characterize the overall quality of peach and nectarine chips from different varieties.

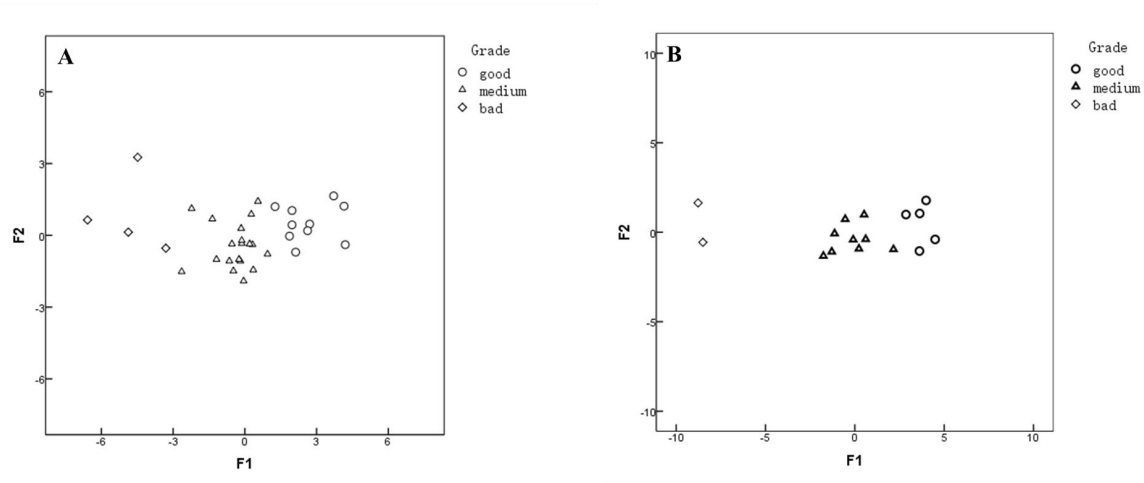
$$Y_{excellent}=-225.299-0.698x_1+1.448x_2+3.115x_3+4.242x_4+1.950x_5$$

$$Y_{medium}=-167.980-0.441x_1+1.250x_2+2.705x_3+3.644x_4+1.538x_5$$

$$Y_{bad}=-135.761-0.002x_1+1.093x_2+2.407x_3+2.388x_4+0.846x_5$$

where x_1 - x_5 were reducing sugar content, rehydration ratio, L value, x_4 crude protein content and explosion ratio, respectively.

Excellent overall quality of peach and nectarine chips was obtained from peach and nectarine varieties, Ruipan 19, Delaifulaika, Dajiubao and other 15 varieties. Medium overall quality was obtained for Ruipan 21, Juhuang, Yanhong and other 28 varieties, while bad overall quality was obtained for Ruipan 20, Huangjinxiu and other varieties. The diversity in quality of peach and nectarine chips within each grade and their relationship is shown in Fig. 7. Here, the score of the first characteristic vector (F1) and second characteristic vector (F2) for all the peach and nectarine chips were represented in the x-axis and y-axis, respectively. For the modeling samples, the discriminant accuracy is up to 100%. For the test samples, only one sample was misjudged. The results showed that the discriminant functions (total discriminant accuracy up to 94.7%) could be used to evaluate and distinguish the overall quality of peach and nectarine peach chips.



A: Modeling samples B: Test samples

Figure 7 Group scatter plot of peach and nectarine chips with different quality on canonical discriminant function

Note: F1 and F2 represent the score of the first characteristic vector and second characteristic vector for all the peach and nectarine chips, respectively. good represents the excellent varieties for processing chips, including Ruipan 19, Delaifulaika, Dajiubao and other 15 varieties; medium represents the medium clutivars for processing chips, including Ruipan 21, Juhuang, Yanhong and other 28 varieties; bad represents the bad cultivals for processing chips, including Ruipan 20, Huangjinxiu and other 4 varieties.

4 Discussion

4.1 Characteristic evaluation indicators

In this study, reducing sugar content, RR, L value, crude protein content and ER were selected as the characteristic evaluation indicators to evaluate the overall quality of peach and nectarine chips. Reducing sugar content was related to both degree of browning during drying and the taste of the products, which could reflect the sweetness. As a substrate of Maillard reaction, reducing sugar content is responsible for the heat-induced browning reaction during the drying processing. A significant correlation ($p<0.05$) between reducing sugar content and L value was found in this study. The inhomogeneous distribution of the reducing sugar content was due to the differences between varieties at cellular tissue level [157].

Rehydration capacity can be influenced by factors including product characteristics, and internal structure of the products [158]. Rehydration capacity might induce many changes in structure and composition of plant tissue, which result in impaired reconstitution properties.

Hence, rehydration capacity can be considered as a measure of the injuries to the material caused by drying treatments [159]. In this study, the use of RR as one of the characteristic evaluation indicators provides a scientific basis for research on quality evaluation of peach and nectarine chips.

Color is an important quality attribute of dried foodstuffs [160]. Discoloration and browning of fruits are the results of various reactions, including Maillard condensation of hexoses and amino components, and pigment destruction [161]. Generally, three colorimetric evaluation parameters (CIELab system), including L value (lightness), a value (green-red hue) and blue value (blue-yellow), are used to describe the changes of color. For PC3 (Table 9), both L value and b value had high weights. However, as the flesh color of peach and nectarine is white to yellow, L value is a more overall evaluation indicator to reflect the color quality of peach and nectarine chips.

Plant material undergo shrinkage and internal tensions during the drying process [162]. Along with the decrease of the moisture content, overall tissue shrinkage, cellular shrinkage and ultimately cell pore collapse take place. Compared with classic drying technology, EPD produces high-quality products with brittle texture, expansion volume and porous microstructure. Therefore, ER was also an important characteristic evaluation indicator.

The porous microstructure of products dehydrated by EPD depended on the film-forming components in the inner plant tissue, such as proteins. Proteins can fully stretch and closely combine with each other in parallel structures. Proteins are capable of forming a continuous matrix during EPD, which could support the porous structure [163, 164]. However, proteins with high content or low degree of organization can limit the ER of products. Therefore, crude protein content viewed as a characteristic evaluation indicator had theoretical significance.

4.2 Application of mathematical analysis methods on the quality evaluation of peach and nectarine chips

FA is not only a technique to reduce the dimensionality by finding a linear combination of variables that explains the variance in the original variables, but also gives prominence to the relationship between the elements. FA has been applied to select the maturity characterization factors for mini watermelon fruit [165], determine parameter of mold inactivation by microwave processing [166] and evaluate the overall quality of Dongzao (*Ziziphus jujube* Mill) [167]. In our study, FA was applied to select five characteristic evaluation indicators from the 17 original evaluation indicators.

AHP can provide quick and automatic qualitative differentiation, in particular when quantification or characterization of specific components of the matrix are not necessary.

AHP has been used to evaluate the quality changes in fruit and vegetable products, such as Jiahua pear [148] and pepper [168]. Based on the results of FA, we obtained the weights of characteristic evaluation indicators by AHP, which allowed us to quantify the impact of the characteristic evaluation indicators on the overall quality of peach and nectarine chips.

DA is also a statistical analysis technique for producing score plots for the analyses [169], and differentiate samples. DA has been used to discriminate varieties of juicy peach (with 100% accuracy) [170] and quality of wheat (with 96% accuracy). In our study, we used the results of DA to characterize the overall quality of peach and nectarine chips from 49 varieties into three different levels, namely, excellent, medium and bad. DA can be used as an important analysis tool for evaluating and distinguishing the overall quality of fruit or fruit products.

5 Conclusion

Forty-nine different peach and nectarine varieties were used to process chips by EPD. Five characteristic evaluation indicators, namely, reducing sugar content, OR, water content, crude protein and ER were obtained by FA. The quality levels in different varieties were effectively classified by discriminant functions obtained by KC and DA. In summary, FA, AHP, KC and DA can be applied to simplify the quality evaluation process and improve its efficiency, with useful application in quality control in the food industry.

Based on the characteristic indicators obtained in Chapter 2 and Chapter 3, both rehydration ratio and expansion ratio are corresponding to texture properties of peach chips. Texture is one of the most important quality attributes of dried fruits and vegetables, reflecting their mechanical and microstructural properties.

In this study, OD and IRD technology are applied prior to EPD (the combined drying processing), which also affect the texture characteristics of peach chips. For OD treatment, a cellular tissue is immersed in a concentrated sucrose solution. OD treatment can provoke changes in macro-micro-and ultrastructure of tissues and water distribution, with several modifications that strongly influence the mechanical behavior and the perceived texture. Nowadays, many attempts have been made to study the texture of dehydrated fruits and vegetables, they can not be extrapolated to the entire drying process, especially for the combined drying technology. Regardless of the temperature, radiation power leads to a significant increase in hardness at some critical moisture content. The significant inconsistency in reported results can be explained by the differences in methodology and different compression strains.

Therefore, the study is aimed to determine the critical moisture content and evaluate the effect of OD pretreatment on the texture characteristics of peach slices at different stages of the combined drying processing.

CHAPTER 4: EFFECT OF SUCROSE CONCENTRATION OF OSMOTIC DEHYDRATION PRETREATMENT ON DRYING CHARACTERISTICS AND TEXTURE OF PEACH CHIPS DRIED BY INFRARED DRYING COUPLED WITH EXPLOSION PUFFING DRYING

This chapter is a modified version of the following research article:

Lyu, J., Yi, J., Bi, J., Chen, Q., Zhou L., Liu, X. and Zhou, M. (2017) Effect of sucrose concentration of osmotic dehydration pretreatment on drying characteristics and texture of peach chips dried by infrared drying coupled with explosion puffing drying. Drying Technology, DOI: 10.1080/07373937.2017.1286670

This study focused on the changes of texture characteristics of peach chips at different stages of a combined drying process, namely explosion puffing drying (EPD) coupled with infrared radiation drying (IRD), in which osmotic dehydration (OD) with different concentrations was applied as the pretreatment. Peach slices were immersed into 100, 300, and 500 g/L sucrose solution for 4 hours at room temperature, respectively, and then peach slices pre-dried to a critical moisture content of 0.5 kg water/kg dry base, which was determined by the effective moisture diffusivity (Deff) curves under IRD treatment at 80 ^{o}C. The peach chips were then dried by using explosion puffing drying (EPD). The sucrose solution with lower concentration (100 g/L) would improve the drying rate of peach slices during infrared drying. However, sucrose solution with higher concentration (500 g/L) might affect water diffusion, resulting in lower drying rate. The changes of texture characteristics of dehydrated peach were ascribed to sucrose uptake during the impregnation step. The content and constitutes of soluble sugars in peach tissue, were significantly affected by OD pretreatment. The results indicated that the combined drying processing, in which OD with appropriate concentration (300 g/L) was applied as pretreatment, could improve the drying characteristics and texture of peach chips.

Key words: osmotic dehydration, critical moisture content, peach, texture

1 INTRODUCTION

China is the leading grower of peach (*Prunuspersica* L.) in the world, in which the production and the area harvested were approximately 11,924,085 tones and 775,000 ha in 2013 (FAO, 2013), respectively.

However, a great loss of peaches during the postharvest period occurs due to a lack of proper postharvest handling and process. The fresh peaches have high perishability due to their high moisture content and the seasonal nature of their production, so peaches should be processed into various products, such as preserved peach and peach chips by using drying technology and processing [171] to prolong their shelf life.

The process of drying is most important, as it has a great effect on the sensory and nutritional characteristics of the final product. However, conventional drying technology e.g. hot-air drying, can exert a great influence to degrade the color, decrease rehydration ratio, reduce volume and decrease porosity of final products. Meanwhile, freeze-drying produces the highest-quality dried foods, but with the longest drying time and the maximum cost. Innovative techniques are permanently being investigated for reducing drying time, saving energy consumption and giving excellent quality for final products. During the past two decades, there has been an increasing interest in combined drying technology because of its several advantages such as faster drying rates, shorter drying times, decreasing energy consumption and improved quality of final products [172].

Compared with conventional drying technology and freeze-drying technology, EPD is an efficient non-fried drying technology with unique advantages [5]. EPD technology which combines the advantages of both hot air drying and freeze drying can provide better functional and organoleptic characteristics of dehydrated food. The principle of expansion by steam is based on a self-vaporization of the moisture contained in the internal of food, which takes place under the influence of a rapid pressure release to atmospheric pressure. Thus, a product with viscoelastic behavior at high temperature expands under the effect of the constraints of the steam generated following decompression, thereby creating a porous structure and a pleasant crispy taste in the product [110]. To reach the critical moisture content of samples, pre-drying methods, such as hot-air drying [12, 18], freeze drying [173, 174] microwave drying [175], and infrared radiation drying (IRD) [5] can be applied prior to EPD. The advantages of infrared radiation are higher drying rate, energy saving, and uniform temperature distribution giving a better quality product [112]. Therefore, EPD coupled with IRD can reduce the drying time and energy consumption, and improve product quality.

Osmotic dehydration (OD) is carried out as a pretreatment before drying processing, removing a part of water contained in a product, giving a better final quality to the product in terms of nutrient retention, texture quality, color stability and also of stability during

storage [176, 177]. During OD processing, the concentration of osmotic solution strongly affects water removal, solid gain, dehydration aspects of the process, as well as drying rate [178]. OD is based on osmosis causing the water movement from low to higher solute concentration region [179]. OD was the most reported pretreatment used prior to drying technology to make available better quality of final products [180, 181]. Baljet et al. [182] identified the optimum processing conditions for OD treatment of peach slices, which indicated that OD could be used as a pretreatment prior to freeze drying or air drying to reduce energy costs and maintain the naturalness of the product. Sahari et al. [183] investigated physicochemical properties of sliced peach during OD pretreatment and dehydration, showing that OD combined with sun-dried samples obtained better quality than sun-dried samples. Germer et al. [184] reported that the concentration of osmotic solution had a strong influence on the sensory quality of sliced peaches which was dried by hot air drying assisted by OD pretreatment. However, little available studies focused on the effect of solution concentration of OD pretreatment on the drying characteristics and texture of peach chips dried by EPD coupled with IRD technology.

Mathematical modeling can help us to understand the heat and mass transfer and thereby be used to predict the moisture content of samples, control drying operation and describe the varied transport phenomena mechanism during drying processing [145, 185]. It should be pointed out that a food product must be suitably prepared for EPD process, particularly as regards its rheological properties, which are mainly determined by the moisture content [6]. Therefore, the main objectives of this study were: 1) determine the critical moisture content symbolically separated IRD and EPD period, 2) evaluate the effect of OD pretreatment with different concentration on the texture characteristics of combination dried peach chips.

2 MATERIALS AND METHODS

2.1 MATERIALS

Firm white Japanese peaches (Dajiubao) with the similar size and maturity were selected for drying experiments. Peaches with an initial moisture content of 8.82 ± 0.47 kg water/kg dry base (d. b.) were hand harvested on 29th July 2015 in Pinggu District, Beijing, and stored at 4 ± 0.5 ^{o}C for at most 5 days.

2.2 SAMPLE PREPARATION AND PROCESSING

In this experiment, OD pretreatment and IRD treatment were used prior to EPD. The detailed description of sample preparation and processing was referred to Lyu et al. [42] with small modification: peaches were cut into 9 mm thick round slices by a Laboratory Slicer (model FA-200, NanhaiDefeng electrothermal equipment Co., Ltd., Guangdong China) after

removing peel and stone. The sliced peaches (320 ± 5.0 g) were steeped into sucrose solution (purchased from Sinopharm Chemical Reagent Co., Ltd., Beijing, China) with different concentration (100, 300 and 500 g/L) for 4 h at room temperature . The weight of the total osmotic solutions was taken 4 times of peach sample weight. Samples without OD pretreatment were treated as the control group. Based on the pre-test, a treatment of IRD (80 ^{o}C, 675 W) was used as pre-drying for the samples which had been wrapped with tissue paper to remove superficial water and sugar until the critical moisture content reached. The critical moisture content was determined based on the results of drying curves of the samples. After the above pre-treated procedure, peach chips were produced by using the experimental EPD equipment system developed by Tianjing Qinde New Material Scientific Development Co. Ltd. (Tianjing, China) [41]. Briefly, pre-treated peach slices were equilibrated at 85 °C for 20 min, then the snuffle valves of the EPD equipment were opened to obtain a rapid decompression (less than 0.2 s) to approximately 4 kPa (absolute pressure) in the puffing chamber. After that, the samples were dried under continuous vacuum at 60 °C until reaching final moisture content (<0.07 kg/kg d.b.) [127].

2.3 Texture characteristics

2.3.1 Hardness and crispness

A spherical probe (P/2.5 s) with a mode of Measure Force Compression was mounted in a texture analyzer (TA. XT 2i/50, Stable Micro Systems Ltd., Surry, UK). The probe was passed through the sample at pretest, test and post-test speeds of 1.0, 1.0, 2.0 mm/s, respectively. The trigger force was set to 100 g, and the trigger type was button. Data acquisition rate was set to 500 pps. The texture properties were derived from the force-deformation curve (force and time). The maximum compression force and the number of peaks in the force-deformation curve of each sample were considered as an indication of hardness and crispness of peach chips, respectively [42].

2.3.2 Volume changes and expansion ratio

The volume of peach slices was measured using a VolscanProfiler (VSP 600, Stable Micro System Ltd., Surry, UK). The expansion ratio (ER) was expressed by using the following equation [41].

$$ER = \frac{V_t - V_0}{V_0} \quad (1)$$

where ER, V_t, V_o referred to expansion ratio, volume of the samples after and before EPD processing (cm^3), respectively.

2.3.3 MICROSTRUCTURE

A scanning electron microscope (SEM S-570, Hitachi Ltd., Tokyo, Japan) at 150 kV accelerated voltage and 10-15 mm working distance was applied to analyze the microstructure characterization of peach slices at different stages of the combined drying processing.

2.4 SOLUBLE SUGARS.

Content of fructose, glucose, sucrose and sorbitol were determined by high performance liquid chromatography (HPLC) with a Lichrosorb-N2H (10 µm) column (Alltech S.A.), according to the description by Nieto et al. [186]. Analyses were carried out on a Refractive Index Detector (Waters 2414). The mobile phases were acetonitrile : water (75 : 25 v/v) and the flow rate was 1.5 mL/min. The data was acquired from a Spectra-Physics integrator.

2.5 DRYING CHARACTERISTICS

The drying characteristics of peach slices during the IRD treatment were carried out at infrared power of 625 W. The samples mass was selected during 10 min with a digital balance (the accuracy of 0.001 g). IRD processing was finished until the equilibrium moisture content researched.

The drying kinetics of peach slices for IRD was determined on the basis of mass losses of samples [187]. The moisture content of dried samples at time t can be transformed to be moisture ratio (MR):

$$MR = \frac{X_t - X_e}{X_0 - X_e} \quad (2)$$

where MR is the moisture ratio, X_0 and X_e are the initial moisture content (kg water/kg dry matter, d.b.) and equilibrium moisture content (kg water/kg dry matter), respectively. X_t is the moisture content (kg water /kg d. b.) at any time.

The drying rate (DR) of peach slices was calculated according to Eq. (3)

$$DR = \frac{X_{t+dt} - X_t}{dt} \quad (3)$$

where DR, t, dt were drying rate (kg water ·kg dry matter $^{-1}$ min^{-1}), time (min), time increment (min), respectively. X_{t+dt} and X_t were the moisture content at t+dt and t (kg

water ·kg dry matter $^{-1}$), respectively. The thin-layer drying models used to describe the drying kinetics of peach slices were shown in Table 11.

2.6 Determination of effective moisture diffusivity

The method was used in the estimation effective moisture diffusivity (D_{eff}) of peach slices at corresponding moisture contents under the IRD conditions based on Fickian equation according to the equations described by Darvishi et al [188], considering a constant moisture diffusivity, infinite slab geometry and uniform initial moisture distribution:

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(-\frac{(2n+1)^2 \pi^2}{4L^2} D_{eff} t\right) \quad (4)$$

where, D_{eff} is the effective diffusivity (m^2/s) and L is the thickness (here half) of layer (m). The Eq. (4) can be simplified by taking the first term of Eq. (5):

$$MR = \frac{8}{\pi^2} \exp\left(-\frac{\pi^2 D_{eff} t}{4L^2}\right) \quad (5)$$

Eq. (5) is evaluated numerically for Fourier number, $F_0 = D_{eff} \times t / 4L^2$, for diffusion and can be rewritten as Eq. (6)

$$MR = \frac{8}{\pi^2} \exp(-\pi^2 F_0) \quad (7)$$

Thus, $F_0 = -0.101 \ln(MR) - 0.0213$ (8)

The effective moisture diffusivity was calculated using Eq. (9):

$$D_{eff} = \frac{F_0}{\left(\frac{t}{4L^2}\right)} \quad (9)$$

where, MR is the moisture ratio, dimensionless; L, the thickness (here half of the layer), m; t, the drying time, s.

2.7 Statistical analysis

All experiments were performed in triplicate, and the results were expressed as mean ± standard deviation (SD). Analysis of variance (ANOVA) was carried out to determine any significant differences in measurements using the SPSS 19.0 statistical software (SPSS Inc.

Chicago, IL, USA) and considering the significant at $p<0.05$. Nonlinear regression analysis, curves and histogram were performed using origin 9.0 (OriginLab, Massachusetts, USA).

3 RESULTS AND DISCUSSION

3.1 DRYING CHARACTERISTICS.

The moisture ratio versus drying time for peaches slices at selected IRD condition with temperature 80 oC and radiation power 675 W was presented in Fig. 8. The curve fitting processes were performed with six well-known thin layer drying models (Table 11). More than 10 well-known thin-layer drying models can be used to describe the drying characteristics of peach slices during IRD processing. Therefore, the more models were compared, the more suitable model would be selected to represent the drying behavior of peach slices.

The coefficient of determination (R^2), reduced chi-square (χ^2) and root mean square error (RMSE) were used to compare the relative goodness of fit of the experimental data (Table 12). The best model describing the drying behavior of peach slices was chosen as the one with the highest R^2 and the least RMSE. The reduced χ^2 was used to determine the goodness of the fit. The lower values of the reduced χ^2 are, the better goodness of the fit is. The randomness of the residues also can be used to evaluate the goodness of the fit. Sometimes the randomness of the residues plays the same role in evaluating the goodness of the fit. Therefore, we can choose RMSE or the randomness of the residues to evaluate the goodness of the fit of the models. For samples without OD, the data were best fitted by Chavez-Mendez model, showing the largest R^2 value (0.99966) and the lowest χ^2 and RMSE values (0.000022 and 0.004509, respectively). For samples treated by 100 g/L sucrose solution, Page model was the most adequate in predicting the moisture transfer owning to the lowest average values of χ^2 (0.000005) and RMSE (0.000088) as well as highest average value of R^2 (0.99993). For samples pretreated by 300 and 500 g/L sucrose solution, R^2 values of Modified henderson and Pabis mode were as high as 0.99996 and 0.99994, respectively, and with the lowest values of χ^2 and RMSE. The different models were selected to predict the thin-layer drying characteristics of peach slices with or without OD pretreatment under IRD process.

The optimal models were used to predict moisture ratio values with drying time and explain the drying behavior of peach slices. If we want to get the relationship between moisture content and operation parameter (e. g. drying temperature, infrared power), the further research and analysis should be done. Thus, for practical applications, the suitability mathematical models should be selected based on the operation parameters.

Table 11 Thin layer drying curve models to describe infrared drying kinetics of peaches

No.	Model name	Models	References
1	Newton	$MR = \exp(-kt)$	Bruce (1985)
2	Page	$MR = \exp(-kt^n)$	Page (1949)
3	Henderson and Pebis	$MR = a\exp(-kt)$	Henderson and Pabis (1961)
4	Two-term model	$MR = a\exp(-k_0 t) + b\exp(-k_1 t)$	Henderson (1974)
5	Modified henderson and pabis	$MR = a\exp(-kt) + b\exp(-gt) + c\exp(-ht)$	Karathanos (1999)
6	Chavez-Mendez	$MR = [1-(1-L_2)L_1 t]^{(1/(1-L_2))}$	Chavez-Mendez et al. (1995)

Note: MR, moisture ratio; t, drying time min;
a, b, c, k, h, empirical constants in the drying models;
k_0, k_1, L_1, L_2, empirical coefficient in the drying models;
n, number constants.

Table 12 Values of the drying constants of different models determined trough regression method for peach slices

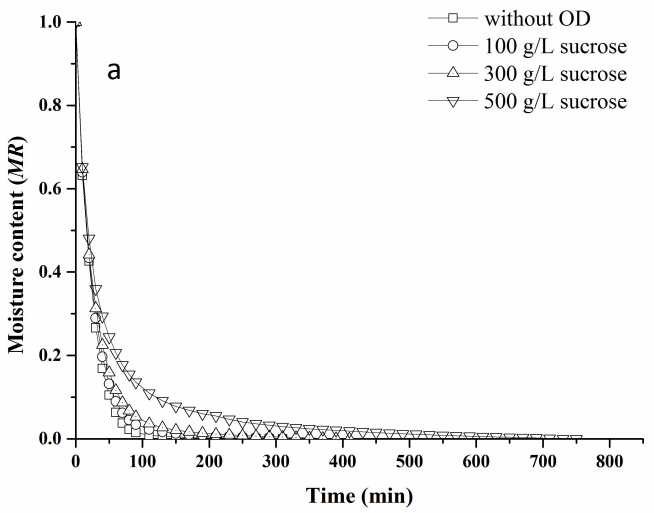
Model No.	Model Name	Conditions	R^2	χ^2	RMSE
1	Newton	Without OD	0.99961	0.000026	0.004956
		100 g/L sucrose	0.99992	0.000005	0.000097
		300 g/L sucrose	0.99616	0.000216	0.023146
		500 g/L sucrose	0.96071	0.00156	0.063200
2	Page	Without OD	0.99961	0.000026	0.004840
		100 g/L sucrose	0.99993	0.000005	0.000088

		300 g/L[1] sucrose	0.99927	0.000041	0.004486
		500 g/L sucrose	0.99584	0.000159	0.00653
3	Henderson and Pebis	Without OD	0.99959	0.000027	0.004956
		100 g/L sucrose	0.99992	0.000005	0.000094
		300 g/L sucrose	0.99653	0.000195	0.004485
		500 g/L sucrose	0.96634	0.001290	0.052860
4	Two-term model	Without OD	0.99954	0.000030	0.004956
		100 g/L sucrose	0.99993	0.000005	0.000078
		300 g/L sucrose	0.99969	0.0000177	0.004432
		500 g/L sucrose	0.99899	0.000039	0.001500
5	Modified henderson and pabis	Without OD	0.99958	0.000028	0.004484
		100 g/L sucrose	0.31824	0.04622	0.647134
		300 g/L sucrose	0.99996	0.000002	0.000011
		500 g/L sucrose	0.99994	0.000004	0.000013
6	Chavez-Mendez	Without OD	0.99966	0.000022	0.004509
		100 g/L sucrose	0.99992	0.000053	0.000095
		300 g/L sucrose	0.99975	0.000014	0.003127
		500 g/L sucrose	0.99941	0.000022	0.000920

Note: OD, osmotic dehydration; R^2, χ^2 and RMSE represent coefficient, chi-squar and root mean square error, respectively.

Peach slices did not exhibit a constant rate period of drying (Fig. 8) but a falling rate of drying

period, which could be explained that the thin-layer of peach slices could not provide a constant supply of water during the whole drying process. What's more, this was also reflected in no or slight case hardening of the products. The moisture ratio (MR) decreased along with the IRD process. Similar results were also reported for the drying of garlic slices [189] and sweet potato slices [190]. Peach slices pre-dehydrated in 500 g/L sucrose solution had a lowest decreasing rate of moisture ratio than peach slices pretreated by sucrose solution of 100 and 300 g/L (Fig. 8a). Revaskar et al. [191] also reported the release of water was found to be fastened with the decrease of the concentration of sucrose solution from 200 g/L to 100 g/L in hot-air drying of osmotic-dehydrated onions. This result was also confirmed by Tan et al. [36]. The lowest drying rate was observed in the samples pretreated by 500 g/L sucrose solution (Fig. 8b) compared with samples without OD treatment and samples pretreated by 100 and 300 g/L sucrose solution. This could be attributed to the binding effect of the osmotic agent with higher concentration that induced more sucrose to diffuse into samples during immersion in the concentrated solutions, causing water removal to be more difficult during the drying processing [36]. It is noteworthy that the drying rate of peach slices pretreated in osmotic solution with 100 g/L concentration was rather similar for the samples without OD. This could be explained by the lower concentration of sucrose solution that might affect moisture diffusion slightly.



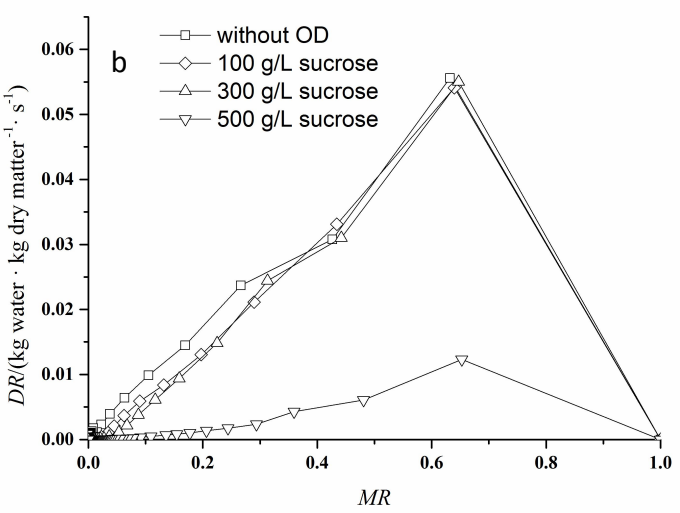


Figure 8 MR versus drying duration curves (a) and drying rates versus MR curve (b) for peach slices pretreated by OD with different concentrations during IRD

Note: MR, moisture ratio; DR, drying rate; OD, osmotic dehydration; IRD, infrared radiation drying

After OD pretreatment, the moisture content for peach slices treated by without OD, 100, 300 and 500 g/L) sucrose solution were 5.77, 5.78, 5.49 and 2.21 kg/kg d.b., respectively. It was clearly shown that the initial moisture content of peach slices pretreated by sucrose solutions with higher concentration (500 g/L) was the lowest (Fig. 9). The osmotic pressure caused by the concentration gradient between the intracellular fluid and solute solution could lead to diffusion of water and solid molecules through semi-permeable membrane to achieve osmotic equilibrium. An increase in solute concentration resulted in an increase in osmotic pressure gradient leading in lower moisture content of samples. A lower dry base value would be observed at the beginning of the drying process, while samples experience water loss and solid gain. Effective moisture diffusivity (Deff) values were found to increase with the decreasing of concentration of sucrose solution. This might indicate that as moisture content decreased, the permeability to vapor increased, the pore structure remained open [192]. However, the binding effect of osmotic agent would cause water removal to be more difficult towards the end. During the IRD processing, a rapid increase in Deff was observed at moisture content of 0.5 kg/kg d.b. (Fig. 9). Lower than the moisture content, the Deff was very low and decreased slowly, which indicated that the speed of water diffusion was very small. It might be speculated that Deff versus moisture content depends on two factors. The first was an initial period in which the rate of water evaporation was almost equal to that from a free liquid surface, while the second was a terminal period when the rate of water evaporation fell on further drying [193]. Thus, the moisture content of 0.5 kg/kg d.b. could be as the critical moisture content separating first and second drying period. Then samples dehydrated by IRD with the moisture content of 0.5 kg/kg d.b. would be further dried by EPD technology.

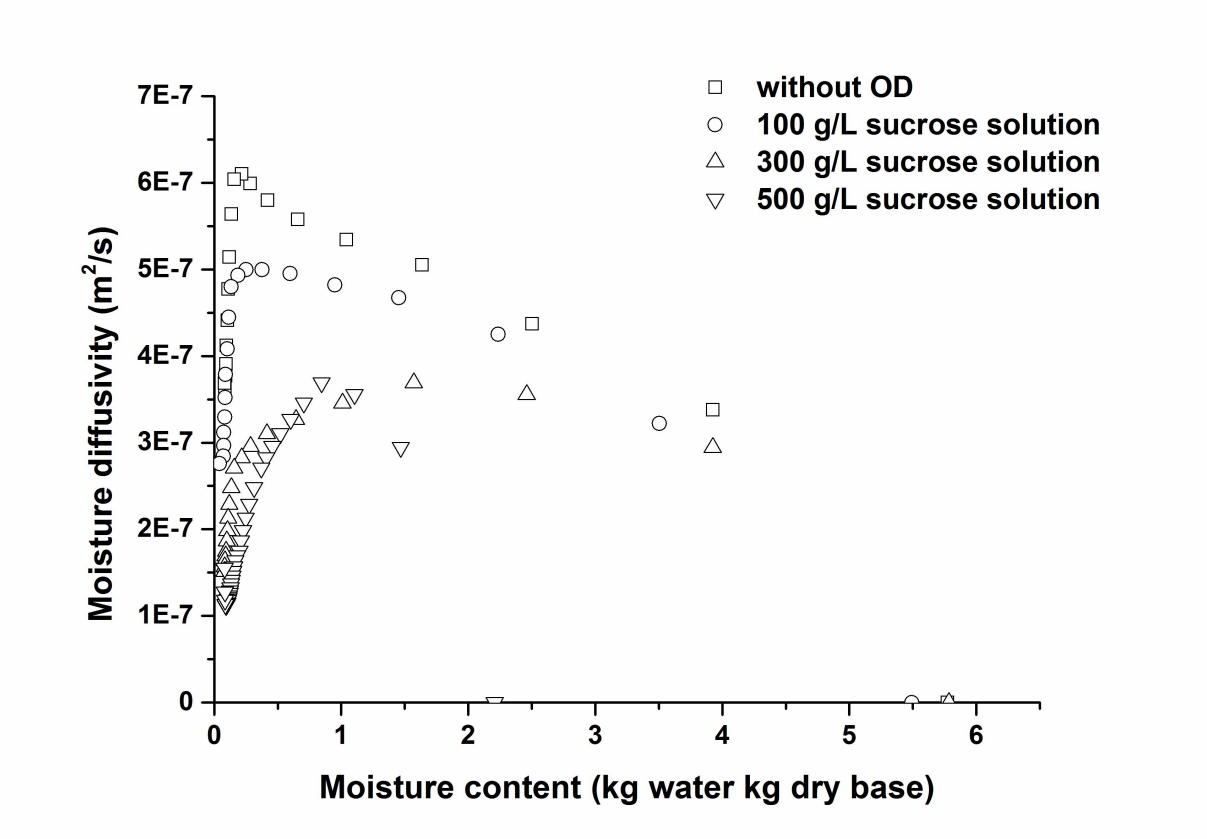


Figure 9 Diffusivity for peach slices pretreated by OD with different concentrations during IRD treatment

Note: OD, osmotic dehydration; IRD, infrared radiation drying.

3.2 TEXTURE CHARACTERISTICS

3.2.1 HARDNESS AND CRISPNESS

Peach chips dehydrated by IR-EPD with OD pretreatment had significantly higher hardness values than samples without OD pretreatment (Table 13). The hardness of the samples pretreated by OD increased as the concentration of osmotic solution increased. The highest hardness value (1534.02 N) was obtained in samples pretreated by 500 g/L sucrose, which was related to the fact that more sugar uptake led to a firm structure, low porosity and loss in elasticity for peach chips [9]. The modest hardness value (847) was found in samples pretreated by 300 g/L sucrose, while, the lowest hardness value (446.64 N) was observed in samples pretreated by 100 g/L sucrose, which had no difference with samples without OD pretreatment. However, products with low hardness would increase in fragility during transportation, and products with high hardness would not be populated in consumers.

Table 13 Effect of OD pretreatment with different concentrations on hardness and crispness of peach chips prepared by IR-EPD

treatments	hardness/N	crispness
Fresh samples	398d ± 1648.56	0d ± 0.00
Without OD	554c ± 3059.62	25b ± 4.26
100 g/L sucrose	447c ± 1662.86	32a ± 3.33
300 g/L sucrose	847b ± 1651.06	25b ± 3.81
500 g/L sucrose	1534a ± 1673.98	20c ± 1.87

Note: Each value is expressed as mean ± SD (n=3).

a, b, c, d, different letters in the same column indicate a significant difference ($P < 0.05$).

OD, osmotic dehydration; IR-EPD, explosion puffing drying coupled with infrared radiation drying.

A decrease in crispness was observed with increasing in sucrose solution concentration (Table 13). The peach slices pretreated with 100 g/L sucrose solution had the highest values of crispness (32.00). Samples pretreated with 500 g/L sucrose solution had the lowest values of crispness (20.20), which might be related to the strong interaction between the hydroxyl groups of peach tissue and high sucrose solution concentration, which improved the strength of cell wall polysaccharides [194, 195]. There was no significant difference between crispness of samples treated without OD and samples treated by 300 g/L sucrose solution. Therefore, appropriated sucrose solution concentrations (300 g/L) could moderately increase the hardness and keep the crispness of peach chips.

3.2.2 Volume changes and explosion ratio

The volume changes at the different stages of the combined drying processing and the explosion ratios (ER) of the peach slices pretreated by sucrose solution with different concentration were shown in Table 14. For all the cases, the volumes of samples were significantly changed after the combined drying procedure. The volumes of samples were decreased after OD and IRD treatment, but increased after EPD. The highest ER (1.12) value was found in peach slices pretreated by 300 g/L sucrose solution. Especially for samples after OD pre-treatment, an increase in concentration of sucrose in the samples was accompanied with a significantly decrease in volume. It could be explained that a lot of water was removed from peaches due to the high osmotic pressure caused by high concentration of sucrose solution [9]. The higher shrinkage of the OD treated samples can be explained by the interlamellar hydroxyl (OH-group) bonding of sucrose molecules having hydroxyl end groups,

which plays an important role in strengthening the sugar impregnated materials [194]. The hydrogen bond also made the tissue rigid and limited the expansion of the cellular structure during drying processing (e.g. IRD) [187]. The results were consistent with Fig.C1, C2, D1, D2, E1 and E2. The volumes of samples dried by EPD were increased due to the instant decompression phase of EPD, in which the samples underwent an irreversible adiabatic transformation. Meanwhile, the partial evaporation of water within the samples induced by the decompression could also create engenders mechanical constraints and porosity structure within the product [6].

Table 14 Effect of OD pretreatment with different concentrations on volume changes and ER of peach chips prepared by IR-EPD

treatments	Without OD	100 g/L sucrose	300 g/L sucrose	500 g/L sucrose
Fresh (cm^3)	2.03Ba ± 0.32	2.06Aa ± 0.08	2.02Ba ± 0.08	2.04Aa ± 0.04
after OD (cm^3)	----	1.94Ba ± 0.07	1.76Cb ± 0.05	1.61Cc ± 0.23
after IRD (cm^3)	0.39Cc ± 0.03	0.40Cc ± 0.03	0.63Db ± 0.03	0.95Ba ± 0.04
after EPD (cm^3)	2.13Ab ± 0.22	1.91Bb ± 0.2	2.27Aa ± 0.1	1.99Ab ± 0.13
ER	1.05 b ± 0.2	0.93 b ± 0.09	1.12 a ± 0.07	0.98 b ± 0.1

Note: Each value is expressed as mean ± SD (n=3).

Values with different letters are significant different $P < 0.05$.

Means with different lowercase letters within a column are significantly different ($P < 0.05$), while, means with different uppercase letters within a row are significantly different ($P < 0.05$).

OD, osmotic dehydration; IR-EPD, explosion puffing drying coupled with infrared radiation drying; IRD, infrared radiation drying; EPD, explosion puffing drying, ER, explosion ratio.

3.3 SOLUBLE SUGAR

OD treatment significantly influenced the sugar composition of peach tissues (Fig. 10). For fresh peach slices, the content of sucrose was the highest, followed by the content of fructose and glucose, also containing little sorbitol, which was consistent with the results reported by Yu et al. [197]. However, sorbitol could not be detected (Fig.10 a) after OD pre-treatment because of the difference in osmotic pressure between intracellular environment and extracellular environment produced by sucrose solution, which might lose certain amount of small molecules (e. g. sorbitol) to the external solution. For all the cases, after the

OD pre-treatment, fructose and glucose content of peach slices decreased with increasing sucrose concentration, however, sucrose content of peach slices increased. For samples without OD pre-treatment and samples pretreated by 100 g/L sucrose solution, fructose, glucose and sucrose content of peach slices increased after IRD treatment, which could be explained that disruption of polysaccharides occurred in cell wall by heating and severe degradation of other ingredients in cell walls resulting from the sugar impregnation step [186]. For peach slices pretreated by 500 g/L sucrose solution, fructose and glucose content also significantly increased after IRD treatment. And the sucrose content of peach slices decreased after IRD treatment, which might contribute to the degradation rate of sucrose was faster than the degradation rate of polysaccharide in tissue. For all the cases, fructose, glucose and sucrose content of tissue was decreased after EPD, which indicated that sucrose might undergo hydrolysis to yield glucose and fructose. Additionally, any glucose and fructose presented in samples would be used in chemical reactions, such as Maillard reaction, during drying [198].

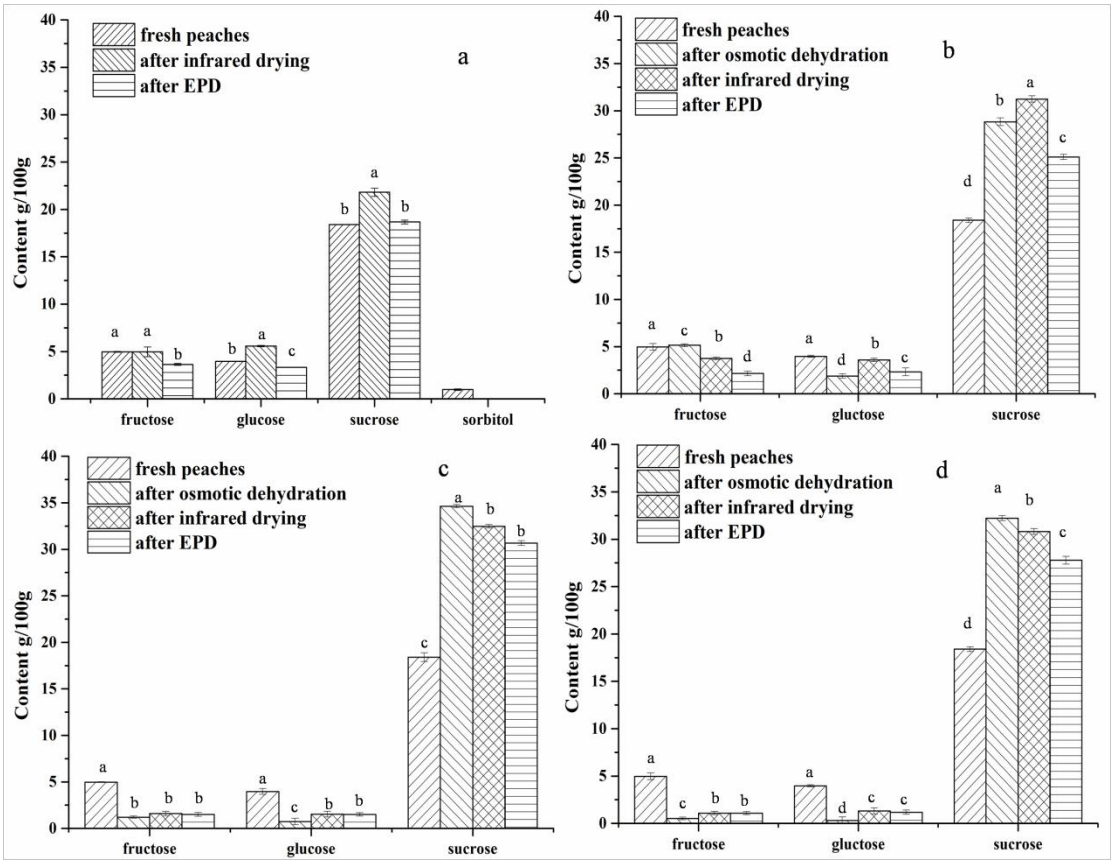


Figure 10 The content and constituent of soluble sugar at different stages of IR-EPD assisted by OD pretreatment with different concentrations. a, samples without OD; b samples pretreated by 100 g/L sucrose solution; c samples pretreated by 300 g/L sucrose solution; d samples pretreated by 500 g/L sucrose solution.

Note: Columns with different lowercase letters are significantly different ($P < 0.05$).

EPD, explosion puffing drying; IR-EPD, explosion puffing drying coupled with infrared radiation drying; OD, osmotic dehydration.

3.4 MICROSTRUCTURE ANALYSIS

Microstructure studies indicated that peach tissues underwent structural changes during the combined drying processing. For the fresh samples, the porous structure was uniform. However, the uniform structure was destroyed and the pore size was decreased associated with the concentration of sucrose solution, according to the infiltration of sucrose solution. Compared with fresh peaches, the intercellular spaces of samples treated by 100 g/L and 300 g/L sucrose solution were occupied by liquid and expanded. The most compact microstructure was obtained in the samples treated by 100 g/L sucrose solution after IRD (Fig.11 C2). For samples pretreated by OD with 500 g/L sucrose solution, a compact microstructure could be observed in Fig.11 E1, showing a great cellular dehydration and sugar penetration in the internal zone of the tissue [199]. With the greatest sugar penetration, peach slices after IRD treatment showed the loose structure, agreeing with the highest volume (Table 14). However, the sugar penetration also inhibited the ER after EPD treatment showing the un-puffed structure (marked with arrows in Fig.11 E3). The microstructure of dried samples pretreated by 300 g/L sucrose solution were uniformed than that of both samples pretreated by 100 g/L and 500 g/L sucrose solution, which could contribute to their relatively better hardness and low crispness. Additionally, un-puffed areas of dense tissue material (marked with arrows) of samples pretreated by 500 g/L sucrose solution were clearly visible in Fig. 11 E2, revealing that some parts of the materials were not puffed. Furthermore, these un-puffed dense areas were expected to fortify the structural strength of dried peach chips treated with 500 g/L sucrose solution, contributing to their relatively high hardness and low crispness [19]. On the other hand, fragments generated by excessive puffing (marked with arrows in Fig. 11 B3 and Fig. 11 C3) could contribute to the relatively low hardness and high crispness. The phenomenon could be explained that the infiltration of sucrose might affect water distribution and structure changes of samples, which would affect the drying characteristics and texture of peach slices.

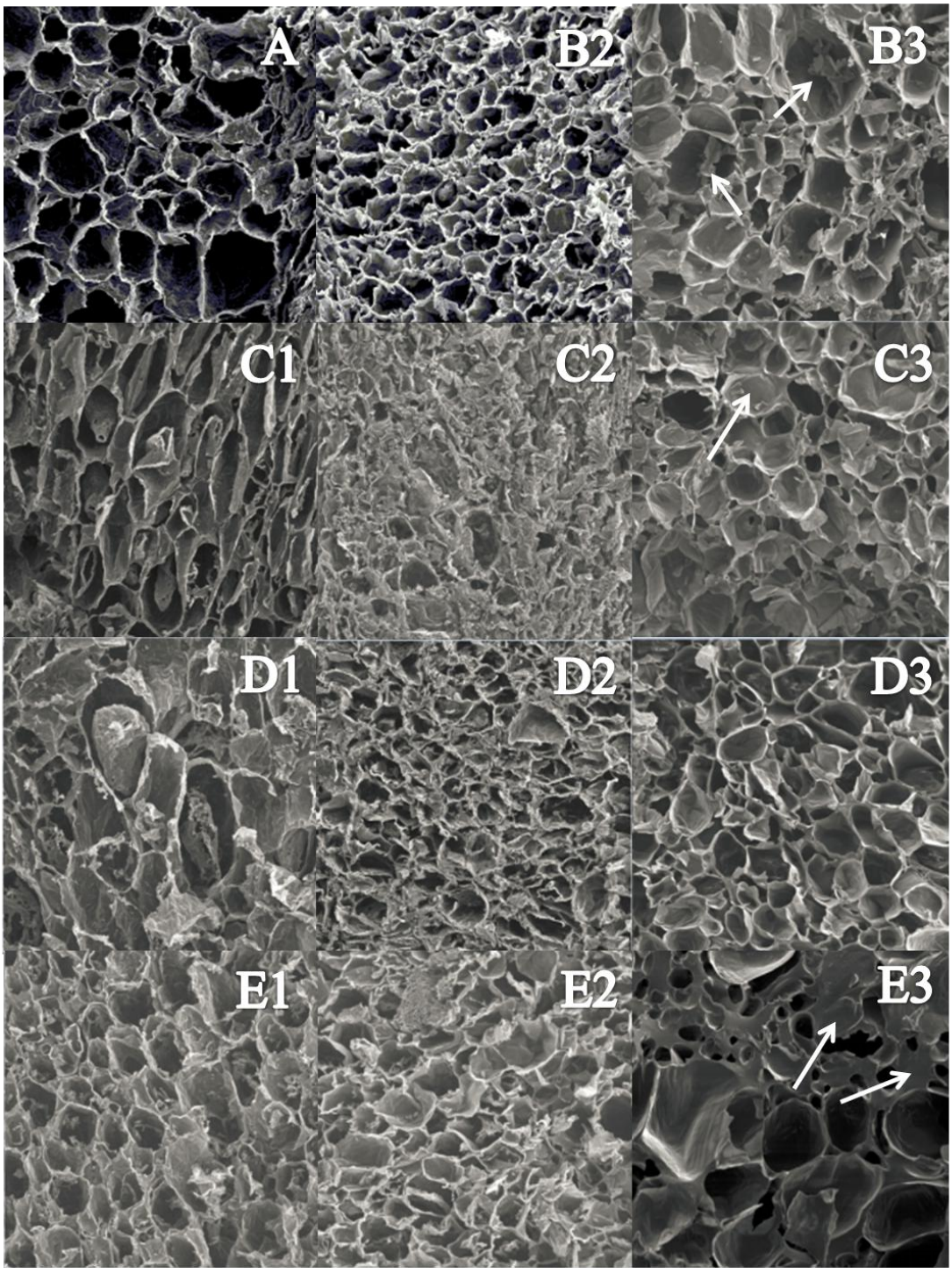


Figure 11 SEM images of peach samples at different stages of IR-EPD assisted by OD pretreatment with different concentrations. A fresh samples; B2 samples dried by IRD without OD pretreatment; B3 samples dried by IR-EPD without OD pretreatment; C1 samples pretreated by 100 g/L sucrose solution; C2 samples dried by IRD with OD pretreatment (100 g/L sucrose solution); C3 samples dried by IR-EPD with OD pretreatment (100 g/L sucrose solution); D1 samples pretreated by 300 g/L sucrose solution; D2 samples dried by IRD with OD pretreatment (300 g/L sucrose solution); D3 samples dried by IR-EPD with OD pretreatment (300 g/L sucrose solution); E1 samples pretreated by

500 g/L sucrose solution; E2 samples dried by IRD with OD pretreatment (500 g/L sucrose solution); E3 samples dried by IR-EPD with OD pretreatment (500 g/L sucrose solution).

Note: OD, osmotic dehydration; IRD, infrared radiation drying; EPD, explosion puffing drying; IR-EPD, explosion puffing drying coupled with infrared radiation drying.

4 CONCLUSION

In this research, the critical moisture content was determined at 0.5 kg/kg d.b. after IRD treatment, with which the dehydration was suggested to be carried on by using EPD technology. Applied OD pretreatments affected the combined drying processing and altered the content and constitute of soluble sugar in peach tissue. The OD pretreatment adversely influenced drying rate, this effect increasing as sucrose concentration of the impregnation solution increased. OD pretreatment also showed a significant effect on texture attributes of peach chips. Peach chips pretreated by 300 g/L sucrose solution showed the good texture characteristics, namely, the modest hardness and crispness, the highest ER and a typical porous structure. It was also deduced that after OD pretreatment with high concentration of sucrose solution (500 g/L), an appreciable reduction in the number of pores of samples took place, slowing the process rate and causing hardening. By considering the drying characteristics and texture attributes of peach, OD pretreatment with appropriate concentration could be applied as an effective treatment prior to the combined drying processing.

When fruit and vegetables are heat treated during processing, some physico-chemical changes of the structural constituents of cell wall and intercellular tissue can be observed. The modification of pectin structure is correlated to texture characteristics of final products. Additionally, plant cell wall, which plays an important role in the physical properties of many fruit and vegetables, is mainly composed by cellulose, hemi-cellulose and pectin. Cellulose and hemi-cellulose are quite stable during common thermal processing. Therefore, Chapter 5 gives a great importance to pectin, according to the fact that its degradation and structural modification can occur during thermal treatment. In Chapter 5, we aim to study the changes of cell wall polysaccharides at the different stages of the combined drying processing, which has a detailed description in Chapter 4.

CHAPTER 5: EFFECT OF PRETREAMENT AND COMBINED DRYING TECHNOLOGY ON WATER STATUS AND CHARACTERISTICS OF WATER SOLUBLE PECTIN OF PEACHES

This chapter is a modified version of the following research article:

Lyu, J., Zhou, L., Bi, J., Liu, X., Zhou, M. and Chen, Q. (2016) Effect of osmotic dehydration and combining drying on water status and characteristics of water soluble pectin on peaches. Manuscript has been submitted to LWT-Food Science and Technology.

Changes of water status and characteristics of water soluble pectin (WSP) of peach were evaluated after different stage of combined drying processing in which osmotic dehydration (OD) was considered as the pre-treatment prior to explosion puffing drying (EPD) - assisted infrared radiation drying (IRD) (IR-EPD). Results showed that the contents of free water and immobilized water decreased to 0 g after the combined drying processing. The content of bound water increased after IRD treatment. The residual pectin methylesterase activity in peach slices was 153.3-179.6% after OD treatment, while, the polygalacturonase were totally inactivated after IRD treatment. The degree of esterification (DE), WSP content and average molar mass (Mw) decreased significantly (from 156.00 to 74.91 mg/g AIR and from 2.28 to 0.49 ×105 Da, respectively) after the combined drying treatment. The neutral sugar of WSP was mainly composed by galactose, arabionse, galacturonic and rhamnose, whose contents decreased after the combined drying treatment. However, OD treatment would slow down the degradation. There was a low peak at 1740 cm^{-1} in Fourier transformed infrared spectrum observed for WSP after IRD and EPD treatment, which might illustrate the degradation of WSP during the combined drying treatment.

Key words: water status; water soluble pectin; degradation; average molar mass; combined drying

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1 INTRODUCTION

Drying is the one of most common technologies for processing and preserving food, especially for fruit and vegetables. Unfortunately, traditional drying methods with high temperature and long drying time easily lead to damage of some product qualities. The combined drying technologies have been proposed in the technology of food dehydration to improve the quality of final products. EPD is based on a self-vaporization of the moisture contained in an item of food when the sudden pressure release to atmospheric pressure [200], which is often used in combination with other drying technologies, such as hot air drying, infrared radiation drying (IRD) and freeze drying. Osmotic dehydration (OD) is the most reported pretreatment applied prior to combined drying, producing the dehydrated products with good quality. The concentration of osmotic solution will strongly affect the water removal and mechanical behavior of plant tissue which is related with the alteration of cell turgor, middle lamella and cell wall resistance [201].

Water, categorizing into bound water, immobilized water and free water[202], is the main component in food matrix, which has a direct influence on the quality attributes of drying products. The immobilized water can be transformed into bound water with lower association degree and free water at different drying conditions. The transformation among different water status can also be affected by fast proton chemical exchange effect with hydroxyl protons on the polysaccharides in the rigid cell wall (e. g. pectin) [203].

Pectin, a complex group of structural heteropolysaccharides containing mostly galacturonic acid units, is present in the primary cell walls and middle lamella of many plants and supports the internal cellular structure [204]. Pectin is susceptible to enzymatic and heat-induced degradation and conformational changes [205]. Pectin methylesterase (PME) and polygalacturonase (PG) are the main enzymes related to change of pectin characteristics. PME can remove the methyl group of pectin, which will be depolymerized by PG, resulting in reducing intercellular adhension and tissue rigidity [206].

Additionally, drying processing can also induce changes in pectin related characteristics, such as galacturonic acid content, arabinogalactan content, paritcle size, degree of esterification (DE) and molecular weight [78, 181]. The enzymatic and/or heat induced changes of pectin will affect the structural-textural characteristics and the capacity of water liberation or binding of dried product [208].

All the above-mentioned processing (OD, IRD and EPD) would affect the transformation of water status and the modification of water soluble pectin (WSP). It can be assumed that the combined drying processing (IRD coupled with EPD), in which OD was used as pretreatment, can affect the characteristics of water status and WSP extracted from peaches.Therefore, the changes of water status and activity of PME and PG under different stages of the

combined drying processing were investigated. The chemical properties of pectin extracted from peaches were characterized in detail. Furthermore, the relationship between water status transformation and WSP properties modification will be discussed.

2 MATERIALS AND METHODS

2.1 MATERIALS

Peaches (*Prunus persica*) with the initial moisture content of 8.82 ± 0.47 kg water/kg dry matter (dry base, d. b.) were hand harvested on 29th July 2015 in Pinggu District, Beijing, China. Peaches were selected with similar size and maturity for further experiments.

2.2 OSMOTIC DEHYDRATION AND DRYING EXPERIMENT

Peaches with high water content could not be directly subjected to EPD. In this experiment, OD and IRD were used prior to EPD in order to lower the moisture content (<35%). The peaches were peeled and the stones were taken out and then the peaches sliced into 9 mm by using a Laboratory Slicer (model FA-200, Nanhai Defeng electrothermal equipment Co., Ltd., Guangdong China) [42].

2.2.1 OSMOTIC DEHYDRATION PRETREATMENT

The peach slices (320 ± 5.0 g) were steeped into sucrose solution (purchased from Sinopharm Chemical Reagent Co., Ltd., Beijing, China) with different concentration (100, 300 and 500 g/L) for 4 h at room temperature. The volume of the total osmotic solutions was taken 4 times of peach samples weight. Peach slices without OD pretreatment were used as the blank control.

2.2.2 INFRARED RADIATION DRYING (IRD)

After OD pretreatment, peach slices (307 ± 3.0 g) with or without OD were dried in a laboratory infrared radiation dryer designed by Senttech Infrared Technology Co., Ltd. (Jiangshu, China). The dryer included six short- and medium infrared lights. The powder of three lights were 225 W, and the others were 450 W. The distance between the lights and the tray was 11 cm. There was a blower which was used to remove the moisture in the air, and the air speed of the blower was 2.11 m/s. A sample tray was set in this dryer and the size of the tray was 31 × 37 cm. Peach slices were arranged onto this tray and dried at 80 ^{o}C, 675 W, 20 -25 % relative humidity, until reached the critical moisture content (0.5 kg/kg, d.b.), which had been determined in chapter 4.

2.2.3 EXPLOSION PUFFING DRYING (EPD)

The above treated samples (150 ± 3.0 g) were placed into the puffing chambers of EPD equipment (Tianjing Qinde New Material Scientific Development Co. Ltd. Tianjing, China), which has been depicted in a previous research [41]. Prior to the pressure release under atmospheric pressure, samples were heated to 90 °C for 20 min under an atmospheric pressure. Then a rapid pressure drop (< 0.2 s) to approximately 4 KPa (absolute pressure) in the processing vessel was attained by opening the decompression valves. Hereafter, the samples were dried under a continuous vacuum at 60 °C until reaching final moisture content (< 0.07 kg/kg d.b.). Each drying processing or treatment was conducted in triplicate.

In addition, fresh peaches, samples after OD treatment, samples after IRD processing and samples after EPD processing were immediately packed into polyethylene bags (20 slices × 9 bags per treatment), and then frozen in liquid nitrogen for further analysis.

2.3 Water low-field nuclear magnetic resonance (LF-NMR)

A low field pulsed NMI 20-Analyst (Shanghai Niumag Corporation, Shanghai, China) with 23 MHz was used to analyze the water status of samples under different stages of the combined drying processing in this experiment. Carr-Purcell-Meiboom-Gill (CPMG) sequence was used to measure the spin-spin relaxation time (T2) [209], which was made with τ-values of 7 μs and 13 μs for 90° and 180° pulse, respectively. The typical pulse parameters were as following: Recycle time = 5,000 ms, echo count =15,000, scan repetitions = 8. The experiment was performed in twice.

2.4 Isolation of water soluble pectin from peaches

Alcohol-insoluble residue (AIR) isolated from the samples and water soluble pectin (WSP) extracted from AIR were obtained as described by Houben et al. [52] with some modification. AIR (5 g) was completely homogenized in 95% ethanol (1 : 6 g/mL) using a mixer (Sanyang mixer, Shanghai, China). After filtration, the residue was homogenized again in 95% ethanol and filtered. Then the combined residue was re-homogenized in acetone (1 : 10 g/mL) before final filtration, followed by drying overnight at 40 °C. The AIR (2 g) was suspended in boiling distilled water (1: 180 g/ml) and stirred for 5 min. The suspension was cooled under running tap water and filtered. The filtrate was dialyzed exhaustively against demineralized water for 24 h. After freeze-drying, WSP extract was obtained.

2.5 Determination of water soluble pectin content

The content of WSP was determined by a colorimetric method with a spectrophotometer (UV-1800, Shimadzu, Kyoto, Japan) as described by Blumenkrantz and Asboe-Hansen [210]. The WSP concentration was expressed as mg/ g AIR.

2.6 Determination of degree of esterification (DE)

The titration method described by Pinheiro et al. [111] was used to determine the degree of esterification (DE). Briefly, WSP (0.2 g) was wetted with ethanol and stirred with distilled water (20 mL) at 40 °C until totally dissolved. The obtained solution was titrated with 0.1 mol/L NaOH (V_1) with phenolphthalein as the indicator. Then a 0.1 mol/L NaOH solution (10 mL) was added to saponify the esterified carboxyl groups of the polymer. The solution was stirred at room temperature for 2 h. Ten milliliters of 0.1 mol/L HCl was added and the excess HCl were titrated with 0.1 mol/L NaOH (V_2).

The value of DE was calculated by the following equation (1):

$$DE\% = \frac{100 \times V_2}{V_1 + V_2} \qquad (1)$$

2.7 Neutral sugar analysis

The content of main neutral sugars (Rhamnose, Arabinose, Galactose and Galacturonic acid) were quantified by using high-performance anion exchange chromatography (HPAEC) with Dionex system (ICS-300 Bio-LC system, USA). Trifluoroacetic acid (TFA) (4 mol/L) was used to hydrolyze pectin samples at 110 °C for 2 h. After cooling, the solution was dried under N_2 and neutralized with 1 M NH_4OH. Samples (10 µL) were injected and eluted by 4 mmol/L NaOH at a flow rate of 0.5 ml/min at 30 °C. The full conditions of equilibration and elution can be found in the report published by Shpigelman et al. [50]. Commercial neutral sugar standards (0.2-20 µg/ml) (Sigma, Shanghai, China) were used as the external standards for identification and quantification.

2.8 Pectin methylesterase preparation and activity assay

The extraction of pectin methylesterase (PME) and its activity assay were performed according to the method proposed by Ly-nguyen et al. [212] with small modification. Samples homogenized and mixed with a 0.2 mol/L Tris-HCl buffer containing 1 mol/L NaCl (pH 8.0; 1:2 w/w) overnight at 4 °C. After filtered using cheesecloth, the supernatant was purified by ammonium sulfate precipitation at 30% saturation and stirred for 30 min. After centrifugation, the supernatant was precipitated again by ammonium sulfate precipitation at 80% saturation. The precipitation containing PME was collected after centrifugation and dissolved in Tris buffer. The activity of PME was measured at pH 7.5 and 30 °C, which was based on carboxyl group titration. A 0.20 mL crude PME solution extracted from samples was mixed with 20 mL of 1% pectin-salt solution (containing 0.1 mol/L NaCl) and incubated at 30 °C. This pectin was purchased from Sigma Chemical (St. Louis, MO, USA). The pH of the solution was adjusted to 7.5 with 0.1 mol/L NaOH. Then 0.025 mL of 0.1 mol/L NaOH were

added and the time to regain pH 7.5 was recorded. The PME units (PMEU) were used to express PME activity calculated by the followed equation (2):

$$PMEU(unit/ml) = \frac{[NaOH] \times V_{NaOH}}{V_{sample} \times t} = \frac{0.1 \times 0.025}{V_{sample} \times t} = \frac{1}{400 \times V_{sample} \times t} \quad (2)$$

Where [NaOH] is NaOH concentration (=0.1 mol/L), V_{NaOH} is the volume of NaOH used (=0.025 mL), V_{sample} is the volume of sample used (=0.20 mL), and *t* is the time (in minutes) needed for pH to return to 7.5 after the addition of NaOH.

2.9 Polygalacturonase preparation and activity assay

The methods of polygalacturonase (PG) preparation and activity assay were referred to Tadakittisarn et al. [213] with some modification. Briefly, samples (10 g) stored at liquid N_2 were homogenized in a blender together with an extracting buffer (50 mL) consisting of 0.02 mol/L sodium phosphate buffer (pH 7.0), 0.02 mol/L EDTA (Ethylenediamine tetra-acetic acid), 1% Triton X-100, 0.02 mol/L L-cysteine HCl and 1 mmol/L PMSF (phenyl methyl sulfonyl fluoride). The supernatant was precipitated with ammonium sulphate, and stored at 4 °C for 4 h. Then the precipitate after centrifuging (20,000 g for 20 min at 4 °C) was dissolved in 0.02 mol/L sodium phosphate buffer (pH 7.0, containing 0.1 mM PMSF). After dialysis and centrifugation (20,000 g for 30 min at 4 °C), the clear supernatant of partially purified enzyme was analyzed for PG activity.

One milliliter of enzyme solution and distilled water (1 mL) were added to the assay medium reagents (0.2% polygalacturonic acid) for 30 min at 37 °C, then the reaction was stopped by addition of DNS (3,5-dinitrosalicylic acid). After boiled in water for 5 min, the solution was diluted and its absorbance was measured at a wavelength of 520 nm with a spectrophotometer (UV-1800, Shimadzu, Kyoto, Japan). One unit of PG was defined by the catalyzation of the hydrolytic cleavage to form 1 nmol/L of galcturonic acid (selected as the standard solution) in 1 s.

2.10 Fourier-transformed infrared spectroscopy (FTIR)

WSP was thoroughly mixed with KBr (1:250, w/w) and pelletized. The infrared spectroscopy (IR) of pectin was recorded by Fourier transform infrared spectra (FTIR) (Bruker Corporation, Karlsruhe, Germany), which could record 64 times with a resolution of 4 cm^{-1} from infrared spectra of the 4000-400 cm^{-1} region [65].

2.11 Weight-average molar mass

Weight-average molar mass (M_w) of WSP was determined by using a high-performance size-exclusion liquid chromatography (HPSEC) apparatus equipped with a TSKgel G-4000PWXL

chromatography column (30 cm × 7.8 cm ID) and three detectors: a multi-angle laser detector (Dawn Heleos II), a UV detector (L-2400) and a differential refractometer (OptilabrEX; Wyatt Technology Corporation, Santa Barbara, CA, USA). The column was eluted with 0.1 mol/L $NaNO_3$, 0.04% (w/v) NaN_3 at a flow rate of 0.5 mL/min at 25 °C. The refractive index increment dn/dc was taken to be 0.135 mL/g. When M_w of WSP was measured, the peak began with the differential peak starting and ended with the laser signal peak. The earlier differential peak appeared and the sooner the laser signal peak ended, the greater the M_w was [65].

2.12 Statistical analysis

The experimental data were assessed using SPSS 21.0 statistical software (SPSS Inc. Chicago, IL, USA) and analyses of variance were conducted by ANOVA procedure. Curves and histogram were performed using origin 9.0 (OriginLab, Massachusetts, USA). All experiments were performed in triplicate.

3 Results and discussion

3.1 The water status in peach measured by LF-NMR

The spin-spin relaxation time (T_2) obtained by LF-NMR spectroscopy can be correlated with free water, immobilized water and bound water, which correspond to different cell compartments, namely, vacuoles, cytoplasm and extracellular, and cell wall, respectively [214]. The free water content gradually decreased from 12.18 g (wet basis) in fresh peaches to 0 g in all samples after EPD treatment (Fig. 12a). OD treatment partially removed free water from fresh peach by immersion of cellular tissue in hypertonic aqueous sucrose solutions. The sucrose solution moved into free space of the tissue while free water came out of the cells. Therefore, a slightly decrease in free water content was observed after OD treatment. However, the drastic decrease in content of free water after IRD treatment was observed in Fig 12a, indicating that a large amount of free water was lost at this phase. These findings suggested that the combined drying processing caused a total loss of mobile water in vacuoles. The changes of immobilized water were more complicated compared with those of free water, due to the complex changes of nutritional ingredients in cytoplasm and extracellular space during the combined drying processing [134]. A decrease in content of immobilized water in the samples after IRD treatment was observed in Fig. 12b. However, an increase in content of immobilized water in the samples treated by 500 g/L sucrose solution after IRD treatment, which may have been due to the shift of free water to immobilized water resulting from the increase in the concentration of carbohydrates (mainly sucrose, glucose and fructose) and the degradation of nutrition ingredients in the cytoplasm [203]. Most of the free water was lost after IRD condition, then the immobilized water under EPD condition would become more mobile and undergo a considerable amount of loss. Due to

the shift of immobilized water to bound water more tightly bound to the polysaccharide in the cell wall (e.g. pectin), a higher content of bound water was observed after OD and IRD treatment in Figure 12c. The increase might also have been due to the accumulation of hydrates in the space between cell wall and plasmalemma [215].

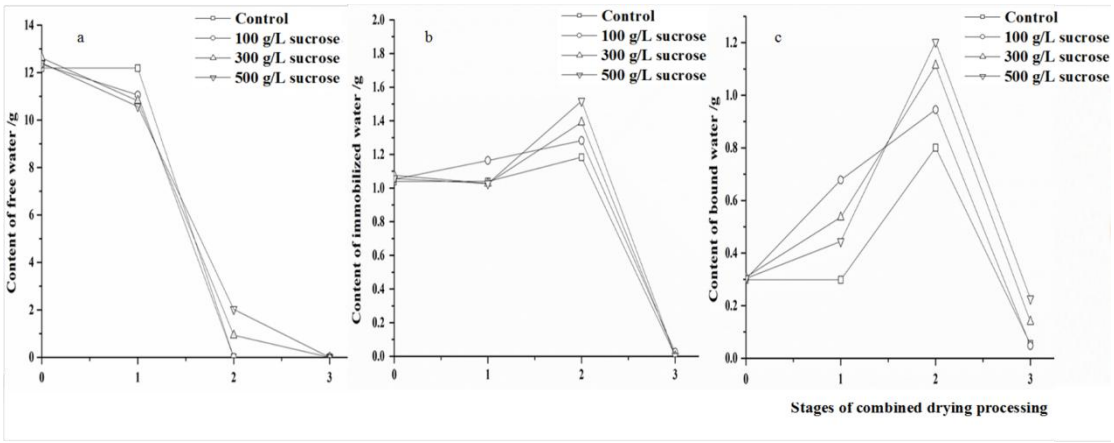

Figure 12 change of different water status at different stages of IR-EPD processing assisted by OD pre-treatment with different concentration

Note: NO. 0-3 on x-axis corresponding to fresh sample, samples after OD, IRD and EPD, respectively.

OD, osmotic dehydration; IRD, infrared radiation drying; DIC, instant controlled pressure drop; IR-EPD, infrared radiation drying coupled to instant controlled pressure drop processing.

3.2 Pectin methylesterase (PME) activity and Polygalacturonase (PG) activity

The changes of pectin methylesterase (PME) and polygalacturonase (PG*)* activity at different stages of the combined drying processing were shown in Figure 13. PME in peach is bound to water-insoluble cell particles and cell walls by an apparent ionic binding [216]. Thus, PME activity increased with the increasing sucrose concentration (Fig 13a), which was attributed to that OD treatment apparently imparted structural and mechanical strength to the tissue and weakened PME binding capacity with cell wall [217]. Moreover, the treatment would affect the selective permeability in the plasma membrane of the fruit, giving rise to diffusion of Ca^{2+} to the cell wall. The increased number of Ca^{2+} would activate PME isoforms, catalyzing the de-esterification of pectin (Table 16) [218]. The residual PME activity in peach slices was 153.3-179.6% and 23-26% after OD and IRD treatment (80 ^{o}C for 60-90 min), respectively. The result was consistent with previous study, which stated that around 12% of PME activity was observed after heated at 80 ^{o}C for 100 min [219]. The possible explanation was that the formation of protein-sugar complex after OD treatment could protect the

denaturation of PME during IRD drying. There was no measurable PME activity observed after EPD treatment, in which samples underwent 90 °C for 20 min (puffing phase) and 65 °C for 1.5-2 h (vacuum phase).

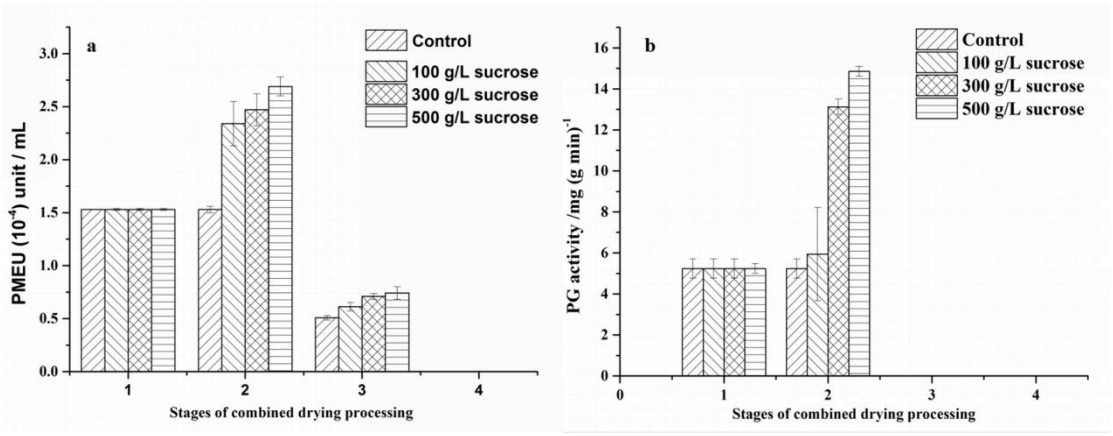

Figure 13 PME activities (a) and PG activities (b) in samples at different stages of IR-EPD processing assisted by OD pre-treatment with different concentration

Note: NO. 1-4 on x-axis corresponding to the progress as fresh sample, samples treated by OD, samples treated by IRD, samples treated by EPD.

PME, pectin mehtylesterase; PG, polygalacturonase; OD, osmotic dehydration; IRD, infrared radiation drying; DIC, instant controlled pressure drop; IR-EPD, infrared radiation drying coupled to instant controlled pressure drop processing.

PG is a cell wall-bound enzyme that catalyzes the cleavage of α-1,4-galacturanosyl linkages producing reducing sugar [71]. The increase in PG activity (Fig. 13b) was observed after OD treatment, which could be explained by the fact that OD treatment weakened the cell wall-bound capacity and released PG activity. DellaPenna et al. [220] reported that two isoforms of PG were presented in fruit: PG1, the heat stable form, could be inactivated at 90 °C, 5 min; PG2, the heat labile form, could be inactivated at 65 °C, 5 min. However, PG was completely inactivated under IRD treatment (80 °C, 60-90 min). It was deduced that the waves produced by IRD possibly penetrated and produced heating inside the fruit, which might alter the PG catalytic subunit [112] and thereby promoted the denaturation of PG. There was no measurable PG activity in sample after EPD treatment (90 °C for 20 min).

3.3 Water soluble pectin content

Water soluble pectin (WSP) contents of samples treated with different sucrose solutions showed similar change trend along the combined drying processing (Table 15). The WSP contents of all samples increased after OD treatment, which could be explained by that the turgor pressure caused by sucrose solution could weaken the hydrogen bonding of pectin in

cell walls [217] and thereby increase the extractable content of WSP. In addition, protopectin solubilization resulting from pectinase may contribute to the increase of WSP contents [221]. However, a significant decrease in WSP content with the increasing concentration of sucrose solution could be observed in samples after IRD treatment, partly attributed to the PME activity existed in the sample during IRD treatment. EPD treatment also resulted in a significant reduction in the content of WSP. This implied that high temperature or instant decompression might cause the heat degradation of WSP.

Table 15 WSP content of peach slices at different stages of IR-EPD processing assisted by OD pre-treatment with different concentration

	Content (mg/g AIR)	control	100 g/L sucrose	300 g/L sucrose	500 g/L sucrose
Sampling in the process	Fresh sample	156.00 ±1.3cA	156.00 ±1.3dA	156.0 0±1.3dA	156.00 ±1.3dA
	Sample after OD	--	167.00 ±1.7aA	178.50 ± 1.3cB	185.60 ± 2.1bC
	Sample after IRD	110.28 ± 2.1aC	100.37 ± 3.1bB	88.97 ± 3.3aA	79.64 ± 2.0aD
	Sample after EPD	90.62 ± 1.3bB	90.08 ± 2.2cA	85.01 ± 1.9bA	74.91 ± 3.0cC

Note: Each value is expressed as mean ± SD (n=3).

Values with different manuscript are significant different $P<0.05$. Means with different lowercase letters within a column are significantly different ($P<0.05$), while, means with different uppercase within a row are significantly different ($P<0.05$).

WSP, water soluble pectin; OD, osmotic dehydration; IRD, infrared radiation drying; EPD, explosion puffing drying ; IR-EPD, infrared radiation drying coupled to explosion puffing drying; AIR, alcohol insoluble residue.

3.4 Degree of esterification (DE) analysis

The DE values of WSP after different treatments were shown in Table 16. OD treatment induced a substantial decrease in the DE value of samples. This decline was increased with the increase of sucrose solution concentration, which might be attributed to the increase in PME activity during OD treatment. PME hydrolyzes the esters present in the pectin backbone, resulting in a partial de-esterification of pectic homogalacturonan [47]. It could be observed

that IRD treatment also resulted in a significant reduction in DE value, which was induced by the residual PME activities after IRD. Moreover, this decline might also result from the modification of pectin structure induced by the increase in reflectivity and transmissivity by infrared radiation energy, as the free water content was decreased during IRD processing [222]. However, the de-esterification was also exhibited in WSP after EPD while no PME activity was detectable, indicating some chemical de-esterification reaction accelerated by high temperature during EPD. In addition, changes in methyl ester content influenced matrix bonding (hydrogen bonds, ionic bonds, ester bonds), which subsequently affected pectin content.

Table 16 Changes in DE value of WSP at different stages of IR-EPD processing assisted by OD pretreatment with different concentration

	DE %	control	100 g/L sucrose	300 g/L sucrose	500 g/L sucrose
Sampling in the process	Fresh sample	75.00±0.9a	75.00±0.9a	75.00±0.9a	75.00±0.9a
	Samples after OD	--	69.00±1.3bA	63.00±0.4bB	57.33±0.3bC
	Samples after IRD	57.85±0.4bA	53.00±0.2cB	51.75±0.4cB	49.85±0.2cC
	Samples after EPD	52.75±0.2cA	48.85±0.3dB	46.25±0.3dB	43.75±0.5dC

Note: Each value is expressed as mean ± SD (n=3).

Values with different manuscript are significant different $P<0.05$. Means with different lowercase letters within a column are significantly different ($P<0.05$), while, means with different uppercase within a row are significantly different ($P<0.05$).

DE, degree of esterification; WSP, water soluble pectin; OD, osmotic dehydration; IRD, infrared radiation drying; EPD, explosion puffing drying ; IR-EPD, infrared radiation drying coupled to explosion puffing drying.

3.5 Neutral sugar analysis

WSP extracted from fresh peaches contained high contents of galactose, arabinose, galacturonic acid and rhamnose (Table 17), together with small amounts of fructose, xylose and mannose (data not shown), which indicated that the main chain of WSP was composed by (1→4) linked α-D-galacturonic acid. It could be deduced that the linear chain of WSP extracted from fresh peaches might be interrupted by α-L-rhamnopyronosyl units bearing some side chains mainly composed of galactose and arabinose residues [52]. The decrease of neutral sugar content along with the increasing sucrose concentration was observed after

OD treatment, which could be explained by the higher activity of PME and PG under higher sucrose concentration. After IRD processing, the contents of galactose and galacturonic acid increased compared with the samples without OD pre-treatment. This could be explained by that OD treatment could slow down the degradation of pectin homogalacturonan [201], which could keep more measurable galactose and galacturonic acid. For all samples, the contents of main neutral sugar were decreased after OD, IRD, and EPD treatment, resulting from polysaccharides hydrolysis, enzymatic reaction or thermal degradation caused by IRD and EPD. Therefore, the combined drying technology had a significant effect on the decrease of neutral sugar content. Interestingly, the highest content of galacturonic acid was found in WSP obtained from peaches slices pre-treated by 100 g/L sucrose solution. This might suggest that OD pre-treatment with appropriate concentration would slow down the degradation of pectin.

Table 17 The main neutral sugar content of WSP obtained from peach at different stages of IR-EPD process assisted by OD pre-treatment with different concentration

	mg/g	rhamnose	arabinose	galactose	glacturonic acid
control	Fresh samples	3.49	51.61	62.80	21.81
	Samples after IRD	1.93	37.33	38.39	15.01
	Samples afterEPD	1.74	28.12	34.64	10.35
100 g/L sucrose	Fresh samples	3.49	51.61	62.80	21.81
	Samples after OD	1.76	36.54	44.69	19.05
	Samples after IRD	0.75	23.16	38.47	16.87
	Samples after EPD	0.88	21.38	33.75	13.70
300 g/L sucrose	Fresh samples	3.49	51.61	62.80	21.81
	Samples after OD	1.45	34.91	42.94	18.60
	Samples after IRD	1.20	21.68	38.94	16.34

	Samples after EPD	0.69	8.27	24.85	12.91
500 g/L sucrose	Fresh sample	3.49	51.61	62.80	21.81
	Samples after OD	0.40	32.20	41.01	17.28
	Samples after IRD	0.16	1.50	32.97	16.66
	Samples after EPD	0.73	4.69	21.71	10.15

Note: Each value is expressed as the original measurement.

WSP, water soluble pectin; OD, osmotic dehydration; IRD, infrared radiation drying; EPD, explosion puffing drying ; IR-EPD, infrared radiation drying coupled to explosion puffing drying.

3.6 FTIR SPECTRA OF WSP

The infrared spectra of WSP with different treatments were depicted in Figure 14. Region from 3600-3200 cm^{-1} was corresponded to the absorption of the inter and intramolecular hydrogen bonding. The stretching characteristic peak was observed in the range of 3000-2800 cm^{-1} for a C-H stretching band [51]. FTIR spectra in the region between 2000-1000 cm^{-1} represented the major chemical functional groups in pectin [223]. The region between 1800-1500 cm^{-1} is of special interest with regards to the evaluation of the degree of esterification.

OD treatment with the sucrose concentration of 100 g/L had no effect on the major absorption of samples. However, transmittance peaks in the range of 1370-1500 cm^{-1} were more outstanding after OD treatment with the sucrose concentration of 300 g/L and 500 g/L, which was related to the symmetric stretching band (near 1400 cm^{-1}) and asymmetric internal vibrations (near 1500 cm^{-1}) of the methyl group in esters [224]. This result stated that the modification effect of OD pretreatment with high concentration (300 and 500 g/L) was higher than the low concentration (100 g/L). Additionally, enzymatic hydrolysis (PME and PG) could contributed to this changes of methyl group. Samples after IRD and EPD treatment exhibited significant band at 1560-1540 cm^{-1}, which was assigned to amide stretching bands of protein and attributed to an increase in uronic acids and pectin [225]. The low but noticeable peak at 1740 cm^{-1} was observed in WSP extracted from peaches after IRD and EPD treatment, suggesting the decrease or loss of carbonyl ester group, which was consistent with the decrease in DE value [226]. The absorptions below 1500 cm^{-1} could not unambiguously be assigned to any particular vibration, since they correspond to complex

interacting vibrating systems [223]. For all samples after IRD and EPD treatment, transmittance peaks at 1100-1000 cm^{-1} were more significant, possibly because of the stretching vibrations of C-OH side groups and the C-O-O glyosidic bond vibration. The changes of FT-IR spectra indicated that the combined drying processing could modify the structure of WSP, especially samples underwent EPD treatment.

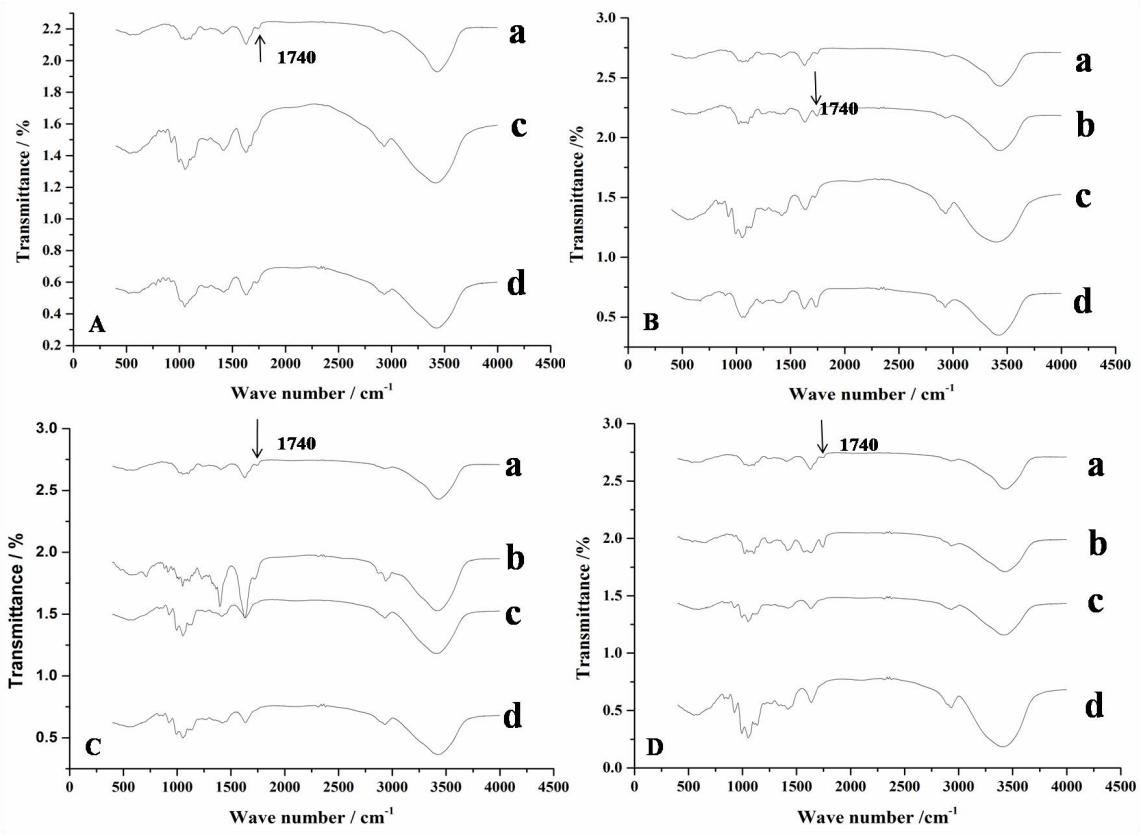

Figure 14 Fourier transform infrared of the 4000-400 cm-1 region of WSP produced by the IR-EPD processing in which OD used as the pretreatment with different concentration (A) control treatment without OD; (B) 100 g/L; (C) 300 g/L; (D) 500 g/L.

Note: a, b, c and d corresponding to fresh sample, samples after OD, IRD and EPD, respectively.

WSP, water soluble pectin; OD, osmotic dehydration; IRD, infrared radiation drying; EPD, explosion puffing drying ; IR-EPD, infrared radiation drying coupled to explosion puffing drying.

3.7 WEIGHT –AVERAGE MOLAR MASS AND ITS POLYDISPERSITY

The average molar mass (M_w) of WSP decreased from 2.28 × 10^5 Da to 0.85 × 10^5, 0.75 × 10^5, 0.57 × 10^5, 0.49 × 10^5 Da after combined drying processing assisted by OD pretreatment with different sucrose concentrations (100, 300 and 500 g/L), respectively (Table 18). This behavior might be attributed to the heat degradation [78]. The OD treatment decreased the M_w of WSP from 1.51 × 10^5 to 1.37 × 10^5 Da with increasing concentration of sucrose solution

from 100 to 500 g/L, and the little polydispersity index (Mw/Mn) was also observed. It was deduced that OD treatment resulting in cell deboning might destroy the connection between the side chains of pectin structure, which led to the decrease of the M_w. In addition, the PG with high activity could catalyze the cleavage of pectin chains, which might lead to depolymerization of WSP chains [218]. The decreases of M_w were also observed in samples after IRD and EPD treatment, in which PG was completely inactivated, suggesting that the heat caused by the combined drying processing might enhance the degradation of polysaccharide. Samples after IRD treatment have the highest amount of polydispersity in each processing although the dispersity was not wide in the samples after EPD treatment. It was worth noting that the effect of EPD on the depolymerization of WSP chains was more efficient than the IRD effect. A major factor accounting for this difference was the severity of the EPD applied. Samples were treated at 80 ^{o}C for 60-90 min during IRD treatment. However, during EPD treatment, samples were treated at 90 ^{o}C for 20 min and then 60 ^{o}C for 1-2 h (vacuum phase), in which samples underwent an additional sudden decompression. At these conditions, the mechanism of WSP depolymerization was not clear yet and might be further studied.

Table 18 Average molar mass (Mw) of WSP and its polydispersity at different stages of IR-EPD processing assisted by OD pre-treatment with different concentratioat different stages of the combined drying processing

		Mw ($\times 10^5$ Da)	Mw/Mn
Control	Fresh samples	2.28 ±0.01aA	1.548±0.28cA
	Samples after IR	1.64 ±0.33bB	2.124±0.38aA
	Samples after EPD	0.85 ±0.14cC	1.872±0.02bA
100 g/L sucrose	Fresh samples	2.28 ±0.01aA	1.548±0.28bA
	Samples after OD	1.51 ±0.01bB	1.257±0.11cC
	Samples after IR	1.34 ±0.20cB	2.167±0.13aA
	Samples after EPD	0.75 ±0.08dC	1.313±0.01cC
300 g/L sucrose	Fresh samples	2.28 ±0.01aA	1.548±0.28cA
	Samples after OD	1.43 ±0.02bB	1.393±0.10bA

	Samples after IR	1.13 ±0.24cC	2.039±0.25aB
	Samples after EPD	0.57 ±0.24dD	1.277±0.29dC
500 g/L sucrose	Fresh samples	2.28 ±0.01aA	1.548±0.28bA
	Samples after OD	1.37 ±0.13bB	1.447±0.11cB
	Samples after IR	1.01 ±0.02cC	2.114±0.17aA
	Samples after EPD	0.49 ±0.28dD	1.329±0.14cB

Note: Each value is expressed as mean ± SD (n=3).

Values with different manuscript are significant different P<0.05. Means with different lowercase letters within a column are significantly different (P<0.05), while, means with different uppercase within a row are significantly different (P<0.05).

Mw, Average molar mass; Mn, number average molecular weight; WSP, water soluble pectin; OD, osmotic dehydration; IRD, infrared radiation drying; DIC, instant controlled pressure drop; IR-DIC, infrared radiation drying coupled to instant controlled pressure drop processing.

4 CONCLUSION

This study showed a combined drying technology to change water status and modify pectin structure extracted from peach slices. The combined drying processing in which OD was considered as the pretreatment was composed by IRD and EPD. OD pretreatment reduced the content of free water and immobilized water. IRD treatment caused a shift from free water to immobilized water and immobilized water to bound water. The activities of PME and PG were promoted by OD pretreatment, however, the heat-induced destruction caused by the combined drying processing inactivated PME and PG completely. The depolymerization of peach pectin was observed after the combined drying processing, which was strongly correlated with the decrease in DE value, main neutral sugar content, WSP content and M_w. According to FT-IR analysis, OD treatment (300 g/L and 500 g/L) could trigger the methyl group's vibration. Additionally, the combined treatment could cause the stretching vibrations carbonyl ester group, carboxylate group and protein. Limitations to this study concern the qualitatively and quantitatively modification of pectin. In addition, peach underwent major chemical modifications through heat-processing and enzyme action. Therefore, a perspective of this work would be correlate the parameters of this combined drying technology and the qualitatively modification of pectin.

It could be deduced that the properties and structures of WSP obtained from peach could be significantly modified by the combined drying processing, in which OD was adopted as pre-treatment and infrared drying was coupled to EPD.

According to the results of Chapter 5, we extracted pectin from fresh peaches, peaches treated by OD, peaches treated by OD followed by IRD and peaches treated by EPD assisted by OD preteatment and IRD pre-dry treatment. The combined drying processing significantly modified the structure of pectin. Multiple lines of evidence indicate a role for pectin in the food and cosmetic industries as well as in pharmaceutique industry.

Pectin can suppress tumor incidence, inhibit cancer cell metastasis and induce apoptosis in cancer cells. However, there is no study focused on the effect of pectin on MM, which is associated with asbestos exposure. In Chapter 6, we investigate the effect of the WSP obtained from Chapter 5 on MM cells and to test if the WSP can induce apoptosis in MM cells.

CHAPTER 6: ANTICANCER ACTIVITY OF PECTIN EXTRACTED FROM PEACH AND PEACH CHIPS DEHYDRATED BY EXPLOSION PUFFING DRYING

In this research, we focused on a cancer associated with asbestos exposure, namely, malignant mesothelioma (MM). We evaluated the ability of water soluble pectin (WSP) extracted from peach and peach chips (Paper 4) to improve mesothelioma therapy. As a surrogate of growth, cell viability was evaluated using the 3-(4,5-dimethylthiazol-2-yl)-5-(3-carboxymethoxyphenyl)-2-(4-sulfophenyl)-2H-tetrazolium (MTS) assay. Propidium iodide (PI) assay was used to evaluate the effect of WSP on cell cycle distribution of mesothelioma cells. Soft agar colony formation assay was performed to assess cellular anchorage-independent growth *in vitro*. Annexin V-PI assays was carried out to detect early and late apoptosis. Results showed that pectin from peach and peach chips could affect MM cell viability, induce apoptosis and decrease colony formation. These observations might be of interest for novel therapeutic developments.

Key words: Malignant mesothelioma, pectin, apoptosis, anticancer activity

1 INTRODUCTION

Malignant mesothelioma (MM) is a neoplastic disease of the pleura strongly associated with exposure to asbestos fibers [227]. The peak of incidence is predicted around 2018 in Western Europe. In the United States, Great Britain and Japan, over 5,000 cases of MM occur annually [228]. MM is usually diagnosed at advanced stage due to its nonspecific clinical and radiographic manifestations [229]. However, with a still increasing incidence due to broad use of asbestos, prognosis of MM is particularly poor. Currently, there is no curative treatment for MM, tumor cells being particularly resistant to chemotherapy. Available treatments have not proven their ability in significantly prolonging survival in comparison to supportive care. To date, chemotherapy based on an association of cisplatin and pemetrexed is the standard treatment used in first line for MM. With a median survival of about 9-12 months, the impact of chemotherapy on the outcome of patients with MM is still limited [230]. Almost half of patients are primary resistant to chemotherapy and almost all of them develop resistance [231]. Thus, attention should be paid to the development of efficient treatments of MM.

Pectin is a class of heterogeneous polysaccharides found in plant cell wall. Pectin, especially for modified pectin, has been reported to inhibit tumor growth, induce apoptosis, suppress metastasis, and modulate immunological responses [91, 82]. The anticancer mechanisms of pectin and modified pectin are correlated with their probiotic activity and immune potentiation. Evidences suggested that pectin and modified pectin may stimulate the immune system [232]. For example, pectin extracted from ginseng can enhance macrophage function and inhibit myeloid-derived suppressor cells to enhance T cell activity [233]. Pectin can be modified by pH, heat, enzymes and radiation usually with low degree of esterification, low molecular weight and other characteristics, which can produce anticancer activity. The pH-modified citrus pectin (MCP) can inhibit tumor growth, angiogenesis and metastases [234]. Leclere et al. [235] reported that citrus pectin modified by heat treatment displayed cytotoxic effects in cancer cells. Kang et al. [236] also produced a citrus modified by irradiation (20 kGy), which was biologically active and inhibited cancer cell growth. In addition, pectin has a broad range of activities depending on the source and the conditions of modification. In this report, pectins extracted from peach and peach chips dehydrated by explosion puffing drying were selected to investigate their anti-apoptotic properties and anticancer activity in malignant mesothelioma cells.

2 MATERIALS AND METHODS

2.1 CELL CULTURE AND DRUGS

The AB1 mouse mesothelioma cells were cultivated in RPMI 1640 medium supplemented with 10% fetal bovine serum (FBS), containing penicillin (100 units/ml) and streptomycin (100 mg/ml) and maintained at 37 ^{o}C in a humidified atmosphere with 5% CO_2.

Pectin extracted from fresh peaches (without peel and stone), peaches treated by osmotic dehydration (OD) treatment, peaches treated by OD - infrared radiation drying (IRD) processing and peaches treated by OD - IRD - explosion puffing drying (EPD) processing were obtained from paper 4 and labeled P1, P5, P6 and P11, respectively. Pectin from citrus was purchased from Sigma.

2.2 Cell viability assay (MTS assay)

As a surrogate of growth, cell viability was evaluated using the 3-(4,5-dimethylthiazol-2-yl)-5-(3-carboxymethoxyphenyl)-2-(4-sulfophenyl)-2H-tetrazolium (MTS) assay (CellTiter 96 Aqueous One Solution Cell Proliferation assay; Promega). Mesothelioma cells (1.6×10^5 cells/ml) were incubated with different concentrations (0-1mg/ml) of pectin1 (P1), pectin 5 (P5), pectin 6 (P6) and pectin 11 (P11). After 48 h of culture, 20 µL of tetrazolium-containing reagent were added to each well in 96-wells plate. After 2 h incubation at 37 ^{o}C in 5% CO_2 humidified air, the plate was analyzed using a colorimetric microplate reader at a wavelength of 490 nm. MTS assay was conducted at least in triplicate.

2.3 Cell cycle analysis

Cell cycle was analyzed by flow cytometry after 48 h of culture in the presence of P1, P5, P6 and P11 with the concentration of 1 mg/ml. Briefly, 3×10^5 cells were trypsinized, collected by centrifugation, washed twice with PBS-10% FBS, and fixed with 70% cold ethanol. After incubation at -20 ^{o}C for at least 1 h, cells were washed twice with PBS-10% FBS and treated for 30 min at 37 ^{o}C with RNAse (50 µg/ml in PBS-0.1% Tween 20; Sigma Aldrich). Then, cells were incubated for 10 min in the dark in propidium iodide (20µg/ml diluted in PBS, Sigma Aldrich) and analyzed by flow cytometry (FACS Aria; Becton Dickinson). Cell doublets were excluded from the analysis using the (FSC-H/FSC-W) gating method. Ten thousand events were collected and analyzed with the FACS Diva Software. The experiment was performed in triplicate.

2.4 Soft agar colony formation assay

Anchorage-independent growth as a characteristic of *in vitro* tumorigenicity was assessed by soft agar clonogenic assay. Briefly, AB1 cells were detached and plated in base agar (1%), supplemented with 2X DMEM, 20% FBS, 100 units/ml antibiotics, 100 units/ml glutamic acid in 24-well plates coated with top agarose (0.7%). 500 cells were incubated for 21 days at 37

^{o}C in humidified incubator and feed with P1, P5, P6 and P1 (1 mg/ml) twice per week. Each experiment was performed in triplicate.

2.5 Detection of apoptosis

Apoptosis was quantified using the Annexin V-phycoerythrin apoptosis detection kit (Becton Dickinson), which labels phosphatidylserine externalized in the early phases of apoptosis. Cells were plated at 1×10^6 per mL in 6-wells plates and treated with P1, P5, P6 and P11 with the concentration of 1 mg/ml. After 24 h of culture, floating and adherent cells were combined, washed twice with cold PBS, suspended in 100 µL of annexin binding buffer (10 mmol/L Hepes, 140 mmol/L NaCl, 2.5 mmol/L $CaCl_2$, pH 7.4), incubated for 15 min at room temperature with 3µL of Annexin V-phycoerythrin and 10µL of propidium iodide (50 µg/ml) and analyzed by flow cytometry (FACS Aria; Becton Dickinson). Ten thousand events were collected and analyzed with the FACS Diva Software.

2.6 Statistical analysis

Statistical significance was calculated using the non-parametric text (Mann Withney) to determine *p* values, and $p < 0.05$ was considered statistically significant.

3 Results

3.1 Effect of pectin on cell viability

In the primary step, the cell viability assay was done in order to determine the most effective time and concentration of pectin. To reach this goal, AB1 cells were treated with different concentration of P1, P5, P6 and P11 (0, 0.2, 0.5, 0.8 and 1.0 mg/ml) for 48 h. Figure 15 indicates that pectin inhibited cell growth after 48 h. Concentration of 1.0 mg/ml of P1, P5, P6 and P11 demonstrated the most effective inhibition of cell viability.

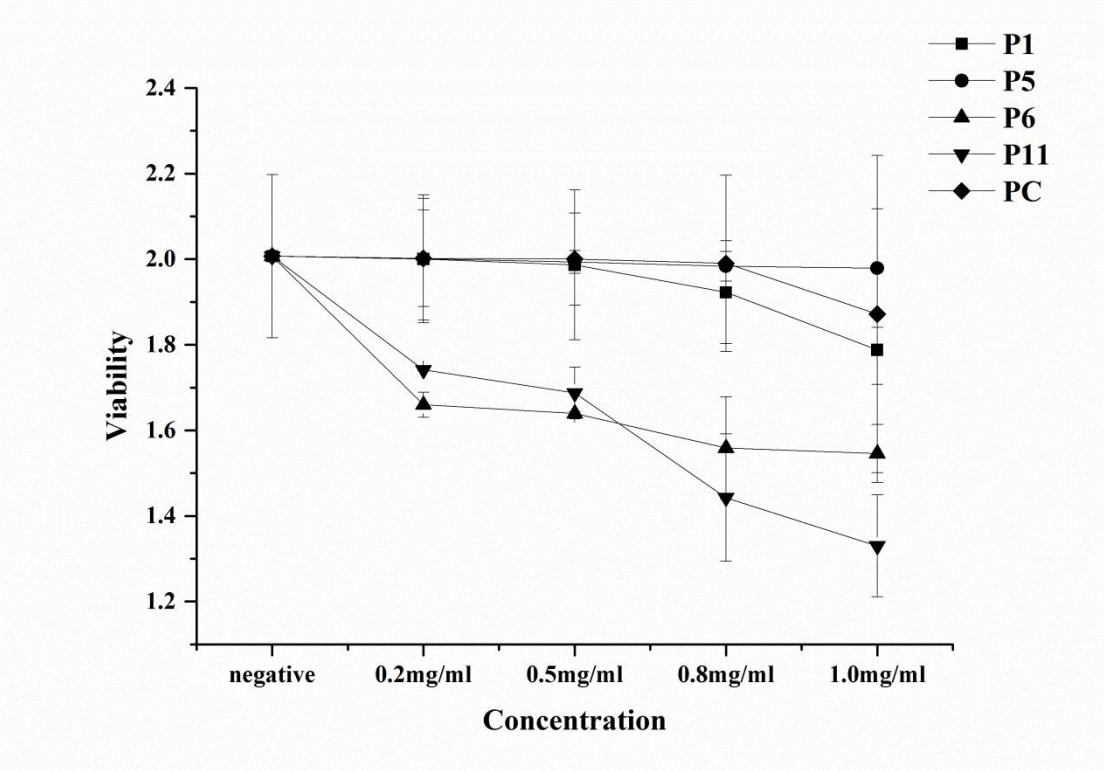

Figure 15 Cell viability of AB1 cells was determined by MTS assay

Note: P1, P5, P6 and P11 represent pectin 1 extracted from fresh peaches (without peel and stone), pectin 5 extracted from peaches treated by osmotic dehydration, pectin 6 extracted from peaches treated by osmotic dehydration - infrared radiation drying and pectin 11 extracted from peaches treated by osmotic dehydration - infrared radiation drying - explosion puffing drying, respectively. PC: pectin from citrus.

3.2 Effect of pectin on cells cycle (PI assay)

To study the effect of pectin on the cell cycle, an analysis of DNA fragmentation was performed by flow cytometry. After cell permeabilization and propidium iodide labeling, cells staining in sub-G1 lose cleaved DNA fragments and are considered to be apoptotic. As shown in Figure 16 (a), the percentages of sub-G1-positive cells was significantly increased in the presence of P1, P5, P6, P11 and PC indicating onset of apoptosis. Flow cytometric analysis of propidium iodide-stained cells further showed that the percentage of cells in S phase was significantly reduced when AB1 cells were cultivated in presence of P1 and P5. The percentage of cells cultivated in presence of P6, P11 and PC were also decreased, but without significance compared with the control group. However, treatment with P1, P5, P6, P11 and PC significantly increased the number of cells in G2/M phase compared to the control. Taken together, these results showed that P1, P5, P6, P11 and PC induce apoptosis and affect the percentages of cells in S and G2/M phases.

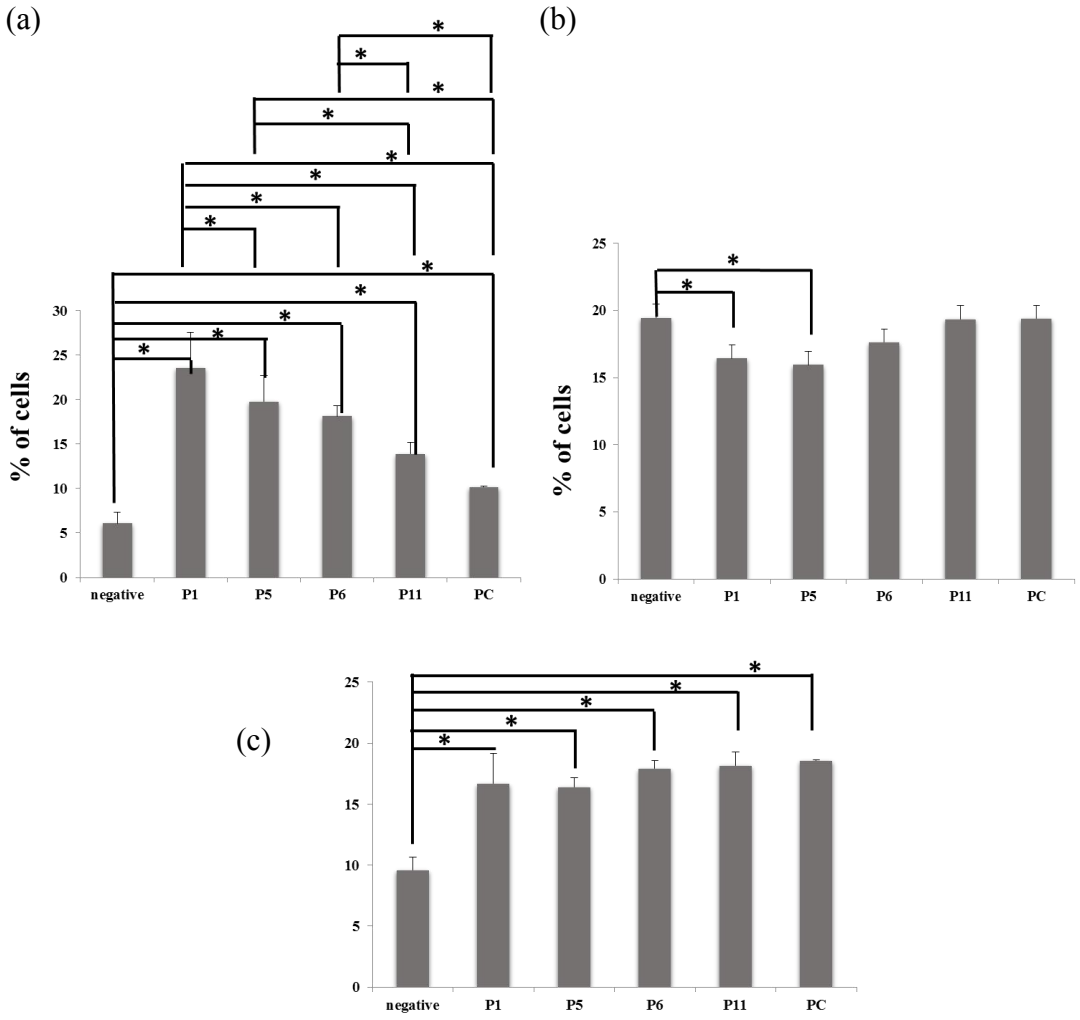

Figure 16 Apoptosis and cell cycle alterations by P1, P5, P6, P11 and P11. (a) Sub-G1 cells were considered to be apoptotic, (b) the percentage of cells in S phase, (c) the percentage of cells in G2/M phase.

Note: Vertical bars show mean + standard deviation from at least three experiments. *p < 0.05. P1, P5, P6 and P11 represent pectin 1 extracted from fresh peaches (without peel and stone), pectin 5 extracted from peaches treated by osmotic dehydration, pectin 6 extracted from peaches treated by osmotic dehydration - infrared radiation drying and pectin 11 extracted from peaches treated by osmotic dehydration - infrared radiation drying - explosion puffing drying, respectively. PC: pectin from citrus.

3.3 CELLULAR ANCHORAGE-INDEPENDENT GROWTH IN VITRO

Anchorage-independent growth is the ability of transformed cells to grow independently of a solid surface, a hallmark of carcinogenesis. The soft agar colony formation assay is a well-established method for characterizing this capability *in vitro* and is considered to be one of

the most stringent tests for malignant transformation in cells [237]. AB1 cells treated with P1, P5 and P11 formed less colonies compared to control (Figure 17). However, P6 and PC treatment did not result in a marked effect in the colony formation assay. These findings further showed that pectin fractions P1, P5 and P11 inhibited anchorage-independent cell growth.

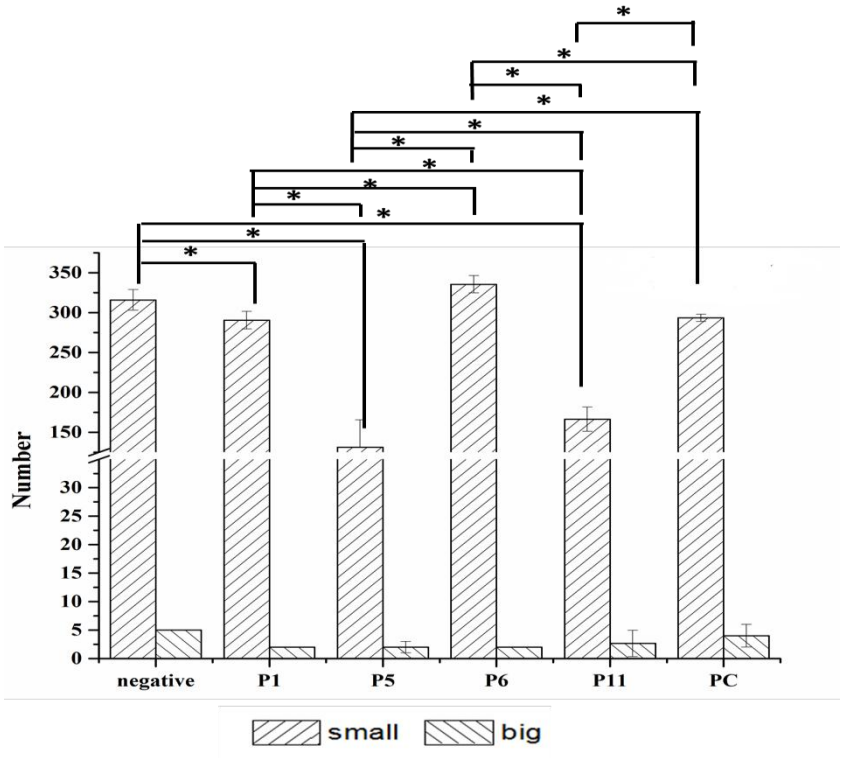

Figure 17 Effect of pectin treatment on colony formation

The number of foci >100 µm was counted. Colonies with foci ≥ 300 µm and foci < 300µm were regarded as big colonies and small colonies, respectively. Vertical bars show mean + standard deviation from at least three experiments. *p < 0.05.

P1, P5, P6 and P11 represent pectin 1 extracted from fresh peaches (without peel and stone), pectin 5 extracted from peaches treated by osmotic dehydration, pectin 6 extracted from peaches treated by osmotic dehydration - infrared radiation drying and pectin 11 extracted from peaches treated by osmotic dehydration - infrared radiation drying - explosion puffing drying, respectively. PC: pectin from citrus.

3.4 APOPTOSIS INDUCED BY PECTIN

Early onset of apoptosis was confirmed by flow cytometry after Annexin V-PI labeling allowing detection of phosphatidylserine externalization. In addition, the loss of plasma membrane integrity is the latest stage of cell death [238]. Annexin V-PI staining was thus performed to quantify early and late stages of apoptosis. The data (Figure 14) showed that

the percentage of cells in the late apoptosis was 8.8% in the untreated group. These percentages significantly increased in presence of P11 and PC. These results confirm that these 2 fractions induce cell death by apoptosis.

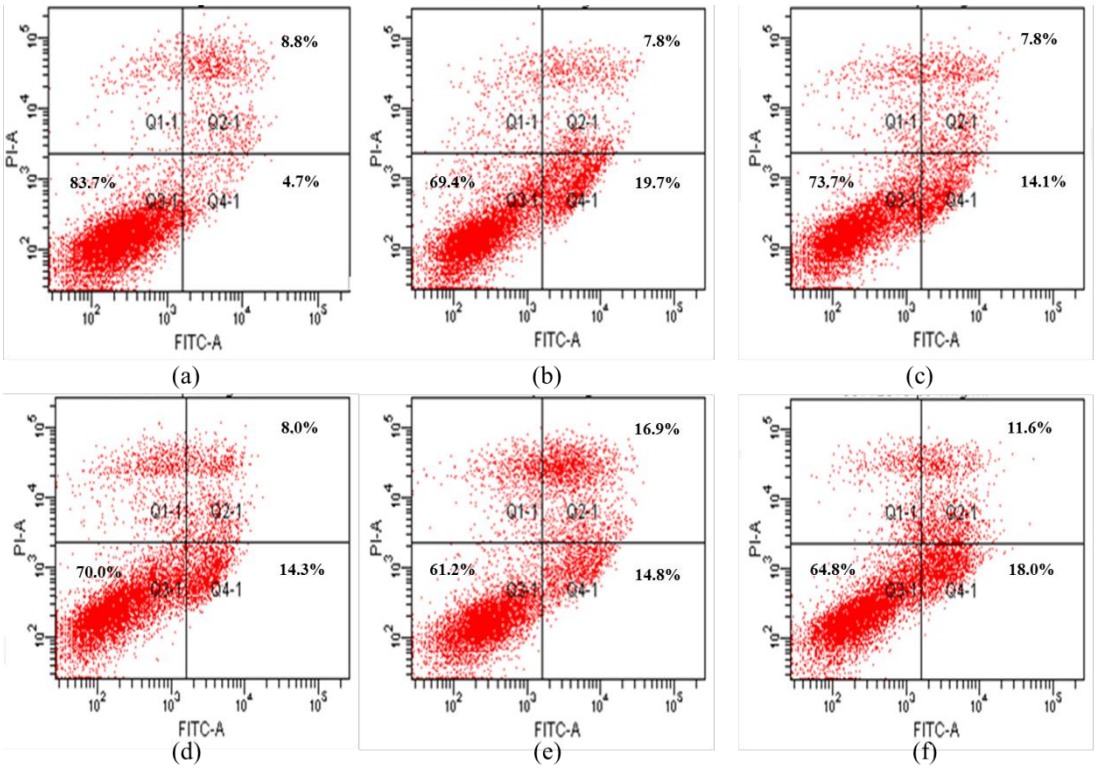

Figure 18 Apoptosis induced by control without treatment (a), P1 (b), P5 (c), P6 (d), P11 (e) and PC (f) was evaluated by flow cytometry after Annexin V- PI labeling.

P1, P5, P6 and P11 represent pectin 1 extracted from fresh peaches (without peel and stone), pectin 5 extracted from peaches treated by osmotic dehydration, pectin 6 extracted from peaches treated by osmotic dehydration - infrared radiation drying and pectin 11 extracted from peaches treated by osmotic dehydration - infrared radiation drying - explosion puffing drying, respectively. PC: pectin from citrus.

4 DISCUSSION

In this study, we analyzed the anticancer activity of peach pectin extracted by different methods as described in chapter 4. These extracts varied in their physico-chemical characteristics and structure. Previous studies indicated that modified pectin has anticancer activity. Leclere et al. [235] reported that citrus pectin modified by heat-treatment displayed cytotoxic effects in cancer cells, which was related to molecules with molecular weight corresponding to low degree of polymerization oligogalacturonic acid. Pectin galactans without arabinose residues and RG-I region of potato induced apoptosis in cancer cells, which can act by binding to and inhibiting the various roles of the mammalian protein

galectin 3 (Gal-3) in cancer progression and metastasis [239]. Jackson et al [92] showed that different fragmentation protocols of pectin could lead to differences in apoptosis-inducing activity of pectin. Fragmented pectin has a cytotoxic effect in androgen-dependent and –independent prostate cancer cells. Evidence also suggested that pectin extracted from natural apples without modification could induce apoptosis in human prostate cancer cells through increasing the release of NO of the mitochondrial apoptosis pathway [82]. In summary, pectin varying in origin, structure and physico-chemical characteristics might possess different anticancer activities. Nevertheless, the mechanism of action of pectin and the pathways involved are still unclear.

Our study indicated that pectin extracted from natural peaches and processed by different treatments caused growth inhibition associated with apoptosis in MM cells. Apoptosis was shown by DNA fragmentation (sub-G1 peak) and confirmed by Annexin V-PI labeling. Perspectives include further characterization of the mechanisms by immunoblotting of proteins involved in apoptosis (e.g. caspase-3 and Bax).

Notwithstanding, our study clearly demonstrates an anticancer effect of pectin obtained by explosion puffing drying. This data opens new prospects for the treatment of MM.

CHAPTER 7: DISCUSSION

1 VARIETY SELECTION

The peach and nectarine varieties used in this study were selected from four main producing areas based on the recent increase in their production throughout Northern China. The fruits harvested from June to September depending on growing conditions (e.g. temperature, rainfall, soil condition). Most of these varieties are only planted in the location where they were harvest.

Our quality evaluation model takes into account that quality differences are related to different varieties, growing conditions and ripening period. Maalekuu et al. [154] assessed the market quality of three colored commercial bell pepper varieties harvested in January and March. Hasanaoui et al. [155] compared the composition, water content and color parameters of fourteen native date varieties from different oases and regions of Morocco, Tunisia and Algeria. Pardo et al. [156] analyzed the physical-chemical and sensory parameters of nine commercial varieties of melon grown in the open air in three different provinces of Spain.

We fully take into account the fact that the quality of peach and nectarine chips is affected by external factors. Therefore, our selection criteria included the size and maturity, the time of year of fruit production, the quality of growing conditions in various regions, and the potential for commercial application. Based on the dataset, the overall quality of peach and nectarine chips allow to separate the 49 varieties into different quality levels.

2 APPLICATION OF MATHEMATICAL ANALYSIS METHODS ON QUALITY EVALUATION

The quality of dried products can be evaluated by complex factors. However, different quality indicators are closely related but relatively independent, which increase the difficulty of quality evaluation. Mathematical analysis methods have been viewed as promising and potential tools to evaluate the overall quality of fruit or vegetable products.

Many evidences have shown that PCA or FA plays an important role in the selection of evaluation indicators. PCA/FA is a simple, non-parametric method for extracting relevant information from confusing data sets, and often used to reduce the dimensionality of data and to solve the multicollinearity problem. The method simplifies the task of obtaining an overview of all the information in the dataset because it is an unsupervised projection method which summaries data by forming new variables as uncorrelated and linear combinations of the original variables [240].

AHP is a prominent and powerful tool to make decisions in situations involving multiple objectives. AHP has the distinct advantage in that it decomposes a decision problem into constituent parts and builds hierarchies of criteria [241]. AHP permits a hierarchical structure of criteria, which gives users a better focus on specific criteria and sub-criteria during weight allocation [242]. Nowadays, AHP has been widely applied in economic, social, political and technological areas to make decision, plan, resolute conflicts and forecast. Thus, AHP will be also widely used in quality control in food industry.

DA is a classification problem, where two or more groups or clusters or populations are known a priori and one or more new observations are classified into one of the known populations based on the measured characteristics. In DA, a parametric method based on normal distribution within each class of samples is used to derive the discriminant function, and the individual within-group covariance matrices are used in calculating distances [243]. Therefore, DA can be used to classify an unknown sample as a member of a particular group.

In our research, PAC or FA is used to reduce the dimensionality of the data from dozens data points (17) to a fewer numbers (5) of dimensions. The effect of this process is to concentrate the sources of variability in the data into the first five PCs. With the five characteristic evaluation indicators obtained by PCA, their relative weights are given to rank decision criteria or alternatives in the use of AHP technique. In addition, the users can evaluate the relative weights of the five characteristic evaluation indicators against the given criteria in sensitively. Based on the scores of overall quality of peach and nectarine chips, the discriminant functions are established by DA technique. We used the results of DA to characterize the overall quality of dehydrated products into three levels, namely, excellent, medium and bad. Furthermore, the more mathematical methods can be applied to simplify and distinguish the overall quality evaluation process, reduce its workload and improve its efficiency in the food industry.

3 Effect of freezing storage and initial water content on texture quality of fruit and fruit chips

Texture of fruit and fruit chips is a complex characteristic resulting from several factors. In this present work, deep-frozen storage, OD treatment and IR-EPD processing can affect the texture characteristics of peach chips. According to the preliminary experiment, sliced peaches were placed in deep-frozen refrigerator at -80 °C for 12 h before undergoing the combined drying processing. The dehydrated peach chips pre-frozen at -80 °C showed the better texture quality than that without deep-frozen storage and that frozen at -20 °C. It is accepted that fast freezing better preserves local structure. Fast freezing can induce the production of a large number of small ice crystals that cause less migration of water and less breakage of cell walls, and consequently less texture deterioration [253].

Freezing at -20 °C with big ice crystals can induce large changes of the cellular structure: cell walls seem to be collapsed [25]. As a consequence of the vacuole rupture, the interaction between cell walls (especially, pectin and cellulose) and cellular contents is facilitated. The combined effect of turgor pressure decrease and cell wall alteration may be responsible for tearing the tissue associated with the bad texture.

After freezing at -80 °C, ice crystals in cell appeared slightly larger than the fresh one. Thus, a large number of small ice crystals may make it possible to maintain cell compartments and the cellular structure in the frozen state. However, the texture difference between the fresh tissue and the frozen/thawed tissue was still high. The freeze storage treatment may result in textural changes leading to food softening, which may be correlated to changes in pectin structure [254].

The cell is the elementary unit within the tissue and its integrity strongly impacts textural quality. Among the many factors involved in fruit texture, the structural integrity of the cell components (cell wall and middle lamella) and cell turgor pressure determined by water content in the vacuoles are the most important. For EPD technology, a food product must be suitably prepared for an expansion process, particularly as regards its rheological properties, which are mainly determined by temperature and the water content. The water content acts on the amount of steam generated during the sudden decompression phase; a large amount of generated steam completely disintegrates the treated product, whereas in the opposite case the products is not well expanded [6]. Consequently, to get the critical moisture content, the drying characteristics of a pre-dry processing should be used prior to EPD processing. Jujube fruit with the initial water content of 0.125 kg/kg d.b. through the fist stage of ambient temperature convection drying [18] is further dried by EPD technology. The initial water content of rehydrated grains can be increased to 0.59 kg/kg d.b. before an expansion procedure [255]. In our study, peach slices are pre-dried with the initial water content of 0.5 kg/kg d.b. and then carried out with EPD treatment. Therefore, the partial drying methods should be carried out to remove partial water of fruit and vegetable based on their characteristics.

4 Effect of pretreatments on the quality of peach chips

OD pretreatment is a water removal technique, which is applied to fruits and vegetables before drying processing. The raw material is placed into concentrated solution soluble solids having higher osmotic pressure are caused by the water and solute activity gradients across the cell membrane, the cell wall and the surface of the tissue [26]. Before further drying, tissue paper is gently used to remove superficial water and the concentrated solution soluble solids, which may affect the drying characteristics of samples during drying processing. OD as a pretreatment to many drying processing can improve nutritional,

sensory, and functional properties of food without changing its integrity. Therefore, OD pre-treatment prior to drying is considered as a minimal heat processing which improves nutritional and sensory properties of dried products.

EPD technology requires low moisture content, therefore, the pre-drying technologies should be used. Hot air drying with the low unit cost is the most commonly used pre-treatment prior to EPD processing. With the emergence of new technologies, IRD technology has been used as an alternative pre-drying method for reducing the moisture content. Meanwhile, IRD can reduce the pre-drying time and bring some positive effects on the volume expansion and texture of fruit chips.

5 EFFECTIVE MOISTURE DIFFUSIVITY DURING THE DRYING PROCESSING

The speed of liquid and/or vapour diffusion is the main mechanism that controls the moisture removing in fruits and vegetables. The variation in moisture diffusivity with moisture content is a complex and system specific function. Deff of a food material characterizes is intrinsic mass transport properties of moisture [118]. Deff is also affected by the composition, moisture content, temperature and porosity of a material. A large knowledge of Deff is necessary for designing and modeling mass-transfer process such as dehydration, adsorption and desorption of moisture during storage. By considering the only diffusion based moisture transfer in thin layer drying, one-dimensional diffusion and Fick's diffusion equation are always used for a simple and accurate analysis of the drying processing.

In this research, the method of slopes is used in the estimation of Deff of peach slices at corresponding moisture contents under IRD processing. An analysis of the falling rate period is carried out to understand the drying kinetics by determination of Deff (Fig. 8 and Fig. 9 in chapter 4). All the figures showed that the drying of peach slices occurred in falling rate period, namely, the liquid diffusion was by the dry wing force controlling the IRD process. However, the indeterminate shapes of samples makes it difficult determine Deff based on Fick's second law. Therefore, finding a suitable geometry to apply the analytic model is an inevitable step in determining the Deff [256]. In case of the peach slice, whose thickness much smaller than radius, it is more appropriate to use the solution of an infinite slab. Thus, the mass transfer is controlled by the thickness of the slice.

Based on the changes of Deff values, we observed an accelerating diffusion speed period existed in the initial period (the moisture content was greater than 0.5 kg/kg d.b.) and a slow falling period in the following period during IRD treatment. Therefore, the moisture content 0.5 kg/kg d.b. Responses the moisture content of the inflexion point where the speed of diffusion transformed into the low one.

6 TEXTURE FORMATION DURING EXPLOSION PUFFING DRYING

Texture plays an important role on the acceptability of dehydrated foods by the consumers. Texture is the result of complex interactions among food components at a microstructural level and at high structural levels as, for instance, the structure of the tissue (cellular orientation, porosity) and the different types of tissues or organs that constitute food materials [244]. What's more, the influence of drying technology and process variables on the texture formation of fruit or vegetable products cannot be ignored. Products prepared by EPD technology always have a crispy texture with high porosity. The parameters involved in EPD processing vary in their importance in terms of the texture of the final products.

The initial water content acts on the amount of steam generated during the decompression phase; a large amount of generated steam completely disintegrates the treated product, whereas in the opposite case the product is not well expanded [245]. During the decompression process, the wet material is subject to high temperature for a short period of time, which allows some moisture contained in the food to vaporize suddenly [196]. The sudden decompression process involves the release or expansion of a gas within a product either to create an internal structure or to expand or rupture an existing structure [15], which mostly depended on the amount of vapor generated, the internal pressure constraints and the pressure drop rate, as well as the viscoelastic behavior of the material. The expansion of a porous structure enhances water diffusivity, which can reduce energy consumption and consequently the manufacturing cost.

The pressure difference (before and after decompression phase) also can affect the texture of products. With the elevated pressure, the increase in explosion ratio can be observed indicating an explosion inside the product creating an important alveolation, which is responsible of the decrease in hardness of products [246]. The sudden decompression causing structural changes by formation of microcavities, requires a sufficient level of steam pressure to assure enough pressure difference to provide the material with adequate rheological properties for expansion. However, excessive pressure difference can cause the disintegration of the product.

In the vacuum drying stage, the final temperature should be low enough to maintain the product structure in an expanded state. The reduction in the final temperature leads to a reduction in heat induced deterioration of sensitive products, which means that the hardening temperature can be exceeded, thus maintaining the structure of the product in an expanded state [246].

In our study, peach slices are dehydrated by IR-EPD, in which OD is performed as the pretreatment. The obtained peach chips have the typical porous structure and a pleasant crispy taste. Therefore, we analyze the texture changes of peach slices during the combined

drying processing to further elaborate the texture formation of peach chips.

In the case of quality properties of dehydrated fruit chips, freeze drying was superior to other drying techniques in preserving physical and nutritional quality. However, freeze drying technology is always inferior in the long drying time and the high production cost. Products prepared by EPD technology have been found to present good quality, such as color, texture and nutritional components [5, 7]. Additionally, the significantly reduction in drying time and reduction cost are also found in the combined drying technology. Therefore, EPD technique with appropriate pretreatment can be recommended for the food drying industry.

7 Modification of pectin during the drying processing

Pectin is a high-molecular weight, biocompatible, nontoxic, and anionic natural polysaccharide extracted from cell walls of higher plants. Pectin is known as functional ingredient, gelling/thickening agent and stabilizer in food industry due to its ability to form aqueous gels. Pectin, regarded as an attractive novel biopolymer material, can be employed in pharmaceutical industry, health promotion and cosmetic applications based on its characters of excellent gelling properties, good biocompatibility and nontoxicity, as well as biodegradability. Moreover, the controlled physical, chemical, and/or enzymatic modification of pectin structure can improve the corresponding physicochemical properties.

Based on the special structure, pectin can be modified by a variety of methods. Enzymatic methods allow regioselective depolymerization under mild conditions. The enzymes fall generally into two groups. In the first group the enzymes responsible for the depolymerization of the HG backbone of pectin, such as pectate lyase, PG, pectin acetyl esterase and PME. The second group of enzymatic activities is directed towards the degradation of RG and side chains. These include rhamnogalacturonase, rhamnogalacturonanan lyase, rhamnogalacturonanan rhamnohydrolase and rhamnogalacturonanan galactohydrolase [56].

The chemical method (e.g. pH) has been proved to be a good tool to generate a large amount of small oligogalacturonides, which can cause growth inhibition correlated with cell death and apoptosis induction in breast cancer cells [248]. In alkaline conditions, an increase of pH increases demethoxylation. Hydrolysis of pectin structure happens in acidic conditions and release different sugar residues present in pectic polysaccharides. The selective degradation of pectin by acid hydrolysis is an important method for revealing the structure of pectin [249]. Researches also showed that pH-modified citrus pectin could inhibit tumor growth, angiogenesis, and metastases [250].

Physical techniques such as high pressure treatment and thermal processing are known to cause major effects on the structure of pectin that may result in depolymerization and

demethoxylation. With high pressure treatment, the linear stiff polymers can easily undergo significant depolymerization compared with the globular and branched structures. During drying processing, the pectin structure can be degraded and modified, which may result in changes of physicochemical properties of the final products and their anticancer activity [19, 91]. Further research can be focused on the relationship between WSP degradation and texture formation of the dehydrated products, which might lead a new perspective on drying technology.

Our data show that degradation and modification of pectin occurred during the combined drying processing. This phenomenon correlates to the heat-induced changes in DE value, M_w and neutral sugar content. The pectin fractions with special characteristics obtained under different stages of the combined drying technology may be used as functional ingredient, gelling/thickening agent, stabilizer and additives in food industry. Moreover, the modified pectin with good features may be considered as novel biopolymer material, and employed in cosmetic application and health promotion. Focusing on the low DE value and M_w, modified neutral sugar groups and bioavailability, application for this modified pectin may be promoted in pharmaceutical industry and cancer therapy. Thus, EPD can be carried out as a potential pathway on modification of pectin.

8 Anticancer activity of pectin

Pectin is a biochemically active compound with known anti-inflammatory, anticarcinogenic and free radical-scavenging properties. Studies suggest that the exposure of malignant cells to pectin and modified pectin induces apoptosis and reduces tumor growth [248, 91]. Evidences show that pectin extracted from citrus is able to induce a prophylactic effect against lung metastasis [89]. Sepehr et al. [82] reported that apple pectin had the potential to induce apoptosis in prostate cancer cells through increasing the release of NO which might be related to the mitochondrial apoptosis pathway. In addition, MCP is believed to adhere to tumor cells through cell surface carbohydrate-binding Gal-3, preventing aggregation of tumor cells and adhesion to normal cells [251].

Studies also suggest that the beneficial effects of pectin are closely related to its structural characteristics. Jackson et al. [92] demonstrated that an ester-based (or related) cross-link in pectin was important for the apoptosis-inducing activity of pectin. Sugar beet pectin treated by alkali, which increased the ratio of RG-I to HG content, increased its bioactivity on tumor cells by inducing apoptosis. The behavior suggested that the neutral sugar side-chains in the RG-I regions was very important for bioactivity [83]. Chen et al. [252] supported the idea that the RG-I domain-rich pectin from potato possessed antiproliferative activity on colon cancer cells. This pectin could induce G2/M phase cell cycle arrest via suppressing cyclin B1 and cyclin-dependent kinase 1 (CDK1).

In this study, the combined drying technology induce the degradation and modification of WSP. Compared with WSP extracted from fresh peaches, the WSP extracted from peach slices at the different stages of the combined drying processing shows the lower DE value and Mw were decrease, which led the better digestion and absorption of organism. Additionally, the stretching vibration of methyl groups, carbonyl ester group and carboxylate group of pectin can result in the modification of side chain groups, which may contribute to the positive effect on reducing metabolism and metastatic stages of cancer or tumor cells.

Although pectin is useful in cancer therapy, the mechanism of induction of apoptosis by pectin is not known. The different extraction protocols and modification methods may alter the structure of pectin and result in differences in its anticancer activity. Therefore, based on further understanding of the structure-activity relationships of the related pectin, pectin fractions can be used as an ingredient in food and pharmaceutical products, which may be beneficial to the prevention and therapy of cancer/tumor.

MM is a highly aggressive and difficult to treat cancer. Treatment options for MM are limited, and the results with conventional therapies have been rather disappointing to this date [105]. Chemotherapy is the only evidence-based treatment for MM patients in good clinical condition. However, the impact of chemotherapy on the outcome of MM patients is still limited, because of its resistant to chemotherapy and radiation. There is a pressing need for more efficacious therapies for MM. Pectin and MP have been indicated to inhibit tumors and cancers, such as liver tumors, colon cancer, prostate cancer, breast cancer and skin cancer. However, no literature surveys cover the important role of pectin and MP in MM suppression. The apoptosis-inducing structure of pectin may be important for bioactivity. The arrangement of neutral sugar might have reduced Gal-3 inhibitory activity and appear to be immunostimulatory [56, 80]. Obviously, pectin and MP have significant anticarcinogenic potential, particularly, by up-regulation cancer cell apoptosis. Therefore, more efforts should be taken to promote the application of pectin in MM therapy.

In this study, WSP is extracted from peach chips dehydrated by EPD technology, which modifies the structure of pectin. We demonstrated that the WSP significantly trigger apoptosis in MM cells. These findings suggest that the WSP has potential to improve MM treatment as a natural product. Improving our understanding of the key pathway in MM and the mechanism of the WSP in apoptosis induction in MM cells will facilitate the development of new treatments. Additionally, an evaluation of the anticancer activity of the WSP must be accompanied by an understanding of the structure of the biologically active pectin.

CHAPTER 8 CONCLUSION AND PERSPECTIVE

1 CONCLUSION

In this study, we apply a novel drying technology, namely EPD to prepare peach and nectarine chips. Based on the requirement of water content, the pre-treatment, such as OD and IRD treatment are used prior to EPD technology in this work.

In the first and second parts of the study, we focus on the quality evaluation of peach and nectarine chips. PCA or FA is used to reduce the diamensionality, and get the first five PCs, which explain the most information of the total variance. Five characteristic evaluation indicators are selected from the connected indicators. AHP is used to get the weights of the five characteristic evaluation indicators and achieve the scores of overall quality of peach chips. Discriminant functions established by KC and DA allow us to divide the peach and nectarine chips into three levels, namely excellent, medium and bad. In summary, the mathematical analysis methods can be applied to simply the quality evaluation process and improve their efficiency in quality control in food industry.

As the results of the first and second parts of this study, ER and RR are the characteristic evaluation indicators, which are related to the texture properties of final products. Therefore, we focus on the changes of texture characteristics peach slices at the different stages of the combined drying processing. In this part, OD treatment with different concentration of sucrose solution was performed prior to IR-EPD treatment. We demonstrated that the changes of texture characteristics of dehydrated peach were ascribed to sucrose uptake during the impregnation step. The sudden decompression stage can significantly affect the formation of porous structure.

Plant cell wall, which plays an important role in the physical properties of many fruit and vegetable products, is mainly made up of complex polysaccharides. Cellulose, lignin and hemi-cellulose are quite stable during common thermal processing. Therefore, a great importance is given to pectin. In the fourth part of this study, we evaluate the changes of characteristics of WSP extracted from peach slices at different stages of the combined drying processing as we did in the third part. The heat-induced destruction caused by the drying process inactivated the activity of PME and PG completely. The depolymerazation of WSP is also observed after the combined drying processing, accompanied by the decrease in DE value, M_w and neutral sugar content. The drying processing also caused the stretching vibrations carbonyl ester group and carboxylate group, which indicate the significant modification of the structure of WSP.

The anticancer activity of the WSP obtained from the fourth part is evaluated in the last part

of this research. The WSP extracted from peach and peach chips could induce apoptosis in MM cells, especially for the WSP extracted from peach chips. The apoptosis pathway and the possible mechanism of the WSP extracted from peach in apoptosis induction in MM cells should be further investigated. This thesis nevertheless contributed to the development of a potential new therapy against MM. In addition, EPD technology can be carried out as a potential pathway on modification of pectin, which will certainly result in more researches on the use of MP.

2 PERSPECTIVE

2.1 VARIETY SELECTION

Based on the production, peaches and nectarine from 49 varieties were collected from June to September in the main producing areas of northern China. Varieties also should be replicated over different seasons and producing areas to establish the quality database of peaches and nectarines. Additionally, the quality of peaches and nectarines can be affected by climate and soil condition.Therefore, the analysis of different varieties should be done in parallel (lasted for three years) to improve the accuracy of discriminant functions obtained in this study.

2.2 QUALITY EVALUATION

PCA, AHP, KC and DA were used in this study to evaluate the variations in the quality of peach and nectarine chips from different varieties. The discriminant functions were also established to distinguish the level of overall quality of peach and nectarine chips. More precise mathematical analysis methods can be investigated and discussed to simplify the evaluation process. For further research, the database of the quality of fresh samples and dried samples should be constructed and used to seek the correlation between fresh samples and dried samples. Thus, we can evaluate the quality of final products through the evaluation indicators of raw material.

2.3 EFFECT OF PRETREATMENT ON TEXTURE QUALITY OF PEACH AND NECTARINE CHIPS

Effect of sucrose concentration of OD pretreatment on drying characteristics and texture properties of peach chips was researched in this study. For sample preparation, freeze storage and IRD treatment were also applied as pretreatments. Freezing rate, time and temperature can affect the formation of crystals, destroy internal microstructure integrity and modify the texture of dehydrated products. IRD power and temperature can influence the moisture diffusion, the permeability and drying rate of the samples. Therefore, more fundamental researches of pretreatments are needed for better design and scale up, so that these technologies can be transferred from laboratory to industry.

2.4 EFFECT OF DRYING PROCESSING ON THE MODIFICATION OF PECTIN STRUCTURE

We evaluated the changes of characteristics of WSP extracted from peach slices at different stages of the combined drying processing. The depolymerazation of WSP was observed after the combined drying processing. Limitations to this study concern the qualitatively and quantitatively modification of WSP. Therefore, a perspective of this study can be correlated the parameters of the combined drying technology, which may induce the quantitatively modification of WSP structure.

2.5 EFFECT OF PECIN ON MALIGNANT MESOTHELIOMA

Our data show that only some fractions of pectin are proapoptotic. The relationship between structural features of pectin and its apoptosis-inducing activity is not known. The elucidation of this mechanism(s) of action of pectin is complicated by 1) the structural complexity of this plant-derived cell wall polysaccharide, 2) the modifications in pectin structure resulting from the process of extraction from plants, and 3) the additional modifications of pectin structure that result from the diverse fragmentation techniques (e.g. pH- , heat, high pressure treatment) used to produce specialized pectin. Therefore, the mechanism of induction of MM cells apoptosis by pectin needs more investigations.

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ANNEXE: LIST OF PEER-REVIEWED ARTICLES

1 PUBLISHED ARTICLES

1.1 FIRST AUTHOR ARTICLES

Lyu, J., Zhou, L., Bi, J., Liu, X. & Wu, X. (2015) Quality evaluation of yellow peach chips prepared by explosion puffing drying. Journal of Food Scinece and Technology **52**(11): 8204-8211

Lyu, J., Liu, X., Bi, J., Zhou, L. & Wu, X. (2016) Research on the quality evaluation for peach and nectarine chips by explosion puffing drying. Scientia Agricultura Sinica **49**(4): 802-812

Lyu, J., Yi, J., Bi, J., Chen, Q., Zhou, L. & Liu, X. (2017) Effect of sucrose concentration of osmotic dehydration pretreatment on drying characteristics and texture of peach chips dried by infrared drying coupled with explosion puffing drying. Drying Technology. (DOI: 10.1080/07373937.2017.1286670)

Lyu, J., Yi, J., Bi, J., Gao, H., Zhou, M. & Liu, X. (2017) Impacts of explosion puffing drying combined with hot-air and freeze drying on the quality of papaya chips. International Journal of Food Engineering. (DOI: 10.1515/ijfe-2016-0250)

Lyu, J., Chen, Q., Bi, J., Zeng, M. & Wu, X. (2017) Drying characteristics and quality of kiwifruit slices with/without osmotic dehydration under short- and medium-wave infrared radiation drying. International Journal of Food Engineering. (DOI: 10.1515/ijfe-2016-0391)

Yi, J., **Lyu, J**., Bi, J., Zhou, L. & Zhou, M. (2017) Hot air drying and freeze drying pre-treatment coupled to explosion puffing drying in terms of quality attibutes of mango, pitaya and papaya fruit chips. Journal of Food Processing and Preservation (DOI: 10.1111/JFPP.13300). (Co first author)

1.2 CO-AUTHOR ARTICLES

Zhang, P., **Lyu, J**., Zhou, L., Bi, J., Liu, X. & Wu, X. (2015) Drying characteristics and energy consumption of peach slices during ultrasound-assisted osmotic dehydration in combination with infrared radiation. Modern Food Science and Technology **31**(11): 234-241

Zhang, P., **Lyu, J**., Bi, J., Liu, X., Zhou, L., Guan, Y. & Xiao, M. (2016) Effect of ultrasound and ultrasound-assisted osmotic dehydration on characteristics by infrared drying. Modern Food Science and Technology 32(11): 197-202

Zhang, P., **Lyu, J.,** Bi, J., Zhou, L., Yi, J. & Wu, X. (2017) Effect of osmotic dehydration on quality of peach chips prepared by explosion puffing drying. Journal of Chinese Institute of Food Science and Technology, 17(1): 69-75

2 ARTICLES SUBMITTED TO A PEER-REVIEWED JOURNAL

MANUSCRIPTS IN PREPARATION

Lyu, J., Zhou, L. & Bi, J. (2017) Effect of osmotic dehydration and combining drying on water status and characteristics of water soluble pectin of peaches. To submit to LWT-Food Science and Technology (revised).