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Evaluation of Drying Kinetics and Physio-Chemical Properties in Development of Golden Prunes

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ABSTRACT Plums are a prominent source of different phytonutrients including phenolic compounds and flavonoids. European plums that are commonly grown in Ontario, Canada are blue-violet in colour, and recently through conventional breeding, a new variety named yellow skinned European plums (YEPs) has been developed. YEPs has its moisture content ranging between 80-90% (wet basis), which makes it prone to microbial spoilage and reduced shelf-life. Hence, drying YEPs to prunes will not only improve its shelf-life but also increase its customer acceptability. In this study, five genotypes of novel Yellow European Plums (YEPs) (Prunus domestica) were dried using conventional dehydration process and its associated effects on YEPs nutritional and physical properties were examined. Dehydration was performed at 50°C, 60°C, and 70°C until a final moisture content of approximately 30% (wet basis) was reached. Eleven drying kinetics models were tested to fit the drying rates of YEPs. Amongst which, Modified Henderson and Pabis and Two-term exponential were observed to best describe the thin layer drying of YEPs. Phenolic content analysis also revealed that dehydration led to an increase of 25-30% in the bioavailability of phenolic compounds in dried YEPS as compared to fresh samples.

Keywords: Dehydration; Antioxidants; Drying Kinetics.

INTRODUCTION Plums (*Prunus)* are considered a rich source of various nutraceuticals and health promoting components such as carbohydrates, vitamins and minerals, which reduces the risk of cardiovascular diseases and improves gastrointestinal health (Arion et al., 2014; Gil et al., 2002). Two cultivars of plums, Japanese (*Prunus salicina*) and European plums (*Prunus domestica*), are the most commonly grown plums in Canada (Okie and Hancock, 2008). However, a new genotype of yellow colored plums has been developed through a novel breeding program. Yellow European Plums (YEPs) have higher amount of phenolic acids, flavonoids, carotenes and antioxidants as compared to traditional blue colored plums (Arion et al., 2014; Tokuşoğlu and Hall III, 2011). Despite their high nutritional value, these fruits have a short shelf life due to their high moisture content (more than 80%). Hence, conversion of plums into prunes using a dehydration process is a critical part of their postharvest handling (Dowling, 2014). Several studies have reported that dried fruits have higher nutrient density, high fiber content, high antioxidant capacity and increased shelf life when compared to fresh fruits, making dehydration of fruits a lucrative business (Kim et al., 2003; Piga et al., 2003).

Various drying methods can be applied in the plum industry for production of prunes. The selection of suitable drying system is a key criterion for production of high quality products, as it can influence the nutritional and physical attributes of the dried products. Several factors such as the type of product, physio-chemical characteristics, energy consumption, and most significantly the cost of dehydration and final quality of the product, affects the choice of drying system to be used (Jangam et al., 2010; Mujumdar, 2014). The most commonly used dehydration methods in plum industry include solar, hot-air, and oven drying. Advanced drying processes include freeze-drying and microwave drying, however, due to their high implementation and maintenance costs, and scale up difficulty, they are utilized less frequently. Commercially, prunes obtained using hot-air drying, are of consistent quality and requires less drying time (Doymaz, 2004; Goyal et al., 2007). Till date no study has been conducted on drying behavior, effect of various drying methods and temperature on physio-chemical characteristics of YEPs. Hence, the objective of this study was to evaluate the drying kinetics of YEPs and optimize the conventional drying process that maintains the nutritional content of the product and result in commercially acceptable golden dried prunes.

MATERIAL AND METHODS

Fruits and chemicals Five genotypes (V99261, V94021, V91074, V98197, and V95141) of YEPs obtained from University of Guelph's breeding program at Vineland (Vineland, Ontario, Canada), were studied. The fresh YEPs were stored at 4°C prior to analysis. Folin-Ciocalteu reagent, sodium bicarbonate (NaHCO3), gallic acid, methanol, 2,2-diphenyl-1-picrylhydrazyl (DPPH), 2,4,6-Tri(2-pyridyl)-s-triazine (TPTZ), formic acid, ammonium formate, and neo-chlorogenic acid were obtained from Sigma Aldrich (Sigma-Aldrich Canada Co., Oakville, Ontario, Canada), whereas methanol, ascorbic acid, sodium acetate, sodium hydroxide (NaOH), hydrochloric acid (HCI), ferric chloride, and chlorogenic acid were obtained from Fisher Scientific (Fisher Scientific Company, Ottawa, Ontario, Canada).

Sample Preparation Plums were washed, patted dry with towel, sliced in half, and pitted. Initial moisture content wet basis (w.b.), was estimated by drying each plum genotype at 105°C for 24 hours in an oven (AACC, 1986). The moisture content was calculated using Equation 1.

$$\text{M. C. (w. b.)} = \frac{M_i - M_f}{M_i} \times 100\% \qquad (1)$$

Where M_i is initial mass of the plum (g), and M_f is the final mass of the plum after 24 hours (g).

Drying Experiment Fresh, pitted plums were dried at three different temperature (i.e. 50°C, 60°C, and 70°C), samples were weighed every 2 hours, until a final moisture content approximately 30% (w.b.) was reached. The dried samples were frozen at -25°C (Insignia Chest Freezer NS-CZ35WH7-C) for 24 hours and then dried in a freeze dryer (Martin Christ GmbH, ALPHA 1- 2LDplus Freeze Dryer) for 48 hours. The freeze-dried samples were then ground into a powder using a blender for further chemical analysis. All drying experiments were conducted in duplicates for statistical validity.

Color Measurements Colour measurements were obtained using a Chroma meter (Konica Minolta SpectraMagic NX, CR-400, Japan). Colour was measured at various points on the skin and pulp of the plum every 2 hours during drying.

Mathematical Modelling Drying behavior of YEPs was determined by fitting the moisture ratio data with thin layer drying models. 11 theoretical and semi-theoretical models were used to evaluate the effect on drying time and temperature on drying kinetics of YEPs (Table 1).

Table 1. Mathematical models used to evaluate the drying kinetics of each plum genotype

Equation No.	Model Name	Equation	References
2.	Newton	$MR = \exp(-kx)$	(Liu and Bakker-Arkema, 1997)
3.	Page	$MR = \exp(-kx^n)$	(Zhang and Litchfield, 1991)
4.	Modified Page	$MR = \exp(-kx)^n$	(Overhults et al., 1973)
5.	Henderson and Pabis	$MR = a\exp(-kx)$	(Henderson and Pabis, 1961)
6.	Modified Henderson and Pabis	$MR = a\exp(-kx) + b\exp(-gx) + c\exp(-hx)$	(Karathanos, 1999)
7.	Logarithmic	$MR = a\exp(-kx) + c$	(Yagcioglu et al., 1999)
8.	Two-term	$MR = a\exp(-k_o x) + b\exp(-k_i x)$	(Sharaf-Eldeen et al., 1980)
9.	Wang and Singh	$MR = 1 + ax + bx^2$	(Wang and Singh, 1978)
10.	Approximation of Diffusion	$MR = a\exp(-kx) + (1-a)\exp(-kbx)$	(Sharaf-Eldeen et al., 1979)
11.	Verma et al.,	$MR = a\exp(-kx) + (1-a)\exp(-gx)$	(Verma et al., 1985)
12.	Two-term exponential	$MR = a\exp(-kx) + (1-a)\exp(-kax)$	(Sharaf-Eldeen et al., 1980)

Where *k* is drying constant; *n, a, b, h, c*, and *g* are the drying coefficients, whose value depends on the model used, *t* is the drying time, and *MR* is the moisture ratio

Preparation of Methanolic Extract Three different extracts samples from fresh, freeze dried and hot-air dried were evaluated. Fresh and dried plum samples were prepared by homogenizing the plum with mortar and pestle. Two grams of the homogenized sample were mixed with 20 mL of methanol. All plum samples were placed in a water bath set at 70°C for 90 minutes. The homogenates were then centrifuged (Servall Enclosed Superspeed Centrifuge, Type SS-4) at 10,000 rpm for 10 minutes, the supernatants were then collected and filtered through a 0.45 μm filter and stored at 3°C for further analysis.

Determination of Total Phenolic content (TPC) TPC for fresh, freeze dried and hot-air dried plums was determined by using modified protocol by Singh et al. (2011). One milliliter of methanolic plum extract was mixed with 7.5 mL double distilled water, 0.5 mL Folin-Ciocalteu reagent, and 1 mL of 7.5% Na_2CO_3 solution. The solution was stored in the dark at room temperature, 23°C, for 90 minutes. The sample absorbance was measured at 765 nm with a spectrophotometer (Thermo Scientific GENESYS 20, Thermo Fisher Scientific, Inc.). The amount of total phenolic content was expressed in terms of gallic acid equivalents (GAE) in mg per g of plum fresh weight (fw) and dry weight (dw).

Scavenging activity on 2,2-diphenyl-1-picrylhydrazyl (DPPH) radicals Free radical scavenging activity of plum extracts on DPPH radicals was measured according to a modified method proposed by Singh et al. (2011) A 50 μL aliquot of methanolic plum extract was added to 1.5 mL DPPH solution (3.94 mg/100 mL methanol). Sample absorption were measured at 517 nm in the spectrophotometer after 20 min, where scavenging activity was calculated using Equation 13.

$$Scavenging\ activity(\%) = 100 \times \left(\frac{Abs_{control}^{517nm} - Abs_{sample}^{517nm}}{Abs_{control}^{517nm}} \right) \tag{13}$$

High Performance Liquid Chromatography (HPLC) Various phenolic components in plum sample such as ascorbic acid, neo-chlorogenic acid, and chlorogenic acid were quantified using HPLC. A Beckman Coulter System Gold instrument (Fullerton, CA, USA). Chromatographic analysis was performed using 32 Karat Software (Version 1.3). Phenolic compounds were separated using a reverse phase C18 Gemini-NX (5μm, 150 mm x 4.6 mm) column (Phenomenex, Inc., Torrance, CA, USA), fitted with a 4mm x 3mm guard column (Phenomenex, Inc., Torrance, CA, USA). The mobile phase was composed of solvent buffer A (10 mM formic acid in water, pH 3.5, with NaOH) and buffer B (5 mM ammonium formate in methanol). The solvent gradient was as follows: 0–1 min 100% buffer A, 1–5 min 0–30% buffer B, 5–8.5 min 30–70% buffer B, 8.5–14 min 70–100% buffer B. UV detection was conducted at 280 nm. A flow rate of 1 mL/minute was used with 20 μL of sample injected. Chromatographic peaks were identified based on retention time of standard compounds of ascorbic acid, neo-chlorogenic acid, and chlorogenic acid. Phenolic compounds in methanolic extracts were quantified using the respective standard curve equations, which were plotted as area under the curve versus concentration (μg/mL).

Statistical analysis A 2x5 full factorial design was used for analysis in which treatment (fresh and hot air dried at 50, 60, 70°C) and genotype were categorical factors. Duncan Tukey Honestly Significantly Different (HSD) test and Analysis of Variance (ANOVA) were conducted to determine the significance of drying method and genotype on colour, phenolic content, scavenging activity, and FRAP. Values reported in this paper for total phenolic content, DPPH, and FRAP are the averages

of three replicates ± standard deviation. Statistical analyses were conducted using JMP software version 11 (SAS Institute Inc., Cary, NC, USA).

RESULTS AND DISCUSSION

Drying kinetics For all plum samples, dried at three different temperatures, moisture ratio (MR) decreased continuously with time whereas, an increase in temperature resulted in an increase of drying rate (Figure 1 and 2). Similar observations for decrease in MR, with respect to time, has been reported by Goyal et al. (2007) during drying of plums in tunnel dryer. During dehydration of plum samples, no constant rate period was observed however only a falling rate period was observed. This suggested that internal mass transfer during dehydration of plums was governed by molecular diffusion (vapor diffusion) or capillary forces, due to which the water from the interior wet regions moves to the surface of the product where it gets evaporated (Toğrul and Pehlivan, 2004). Drying at 50°C for all the samples, resulted in longer drying time as compared to one dried at 60°C and 70°C. For some genotypes (V97261, V91074, V95141), no significant difference in drying time for samples dried at 60°C and 70°C was observed. This variation in time can be attributed to inherent dissimilarity in their physio-chemical characteristics and in their internal tissue structure, which affects the presence of water pockets in the sample.
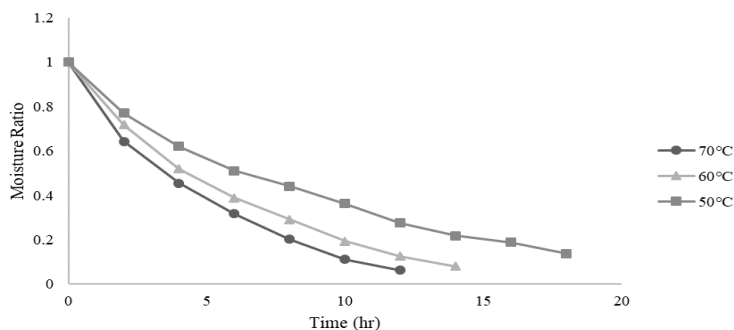

Figure 1. Effect of drying temperature 70°, 60°, and 50°C on Plum Genotype V94021
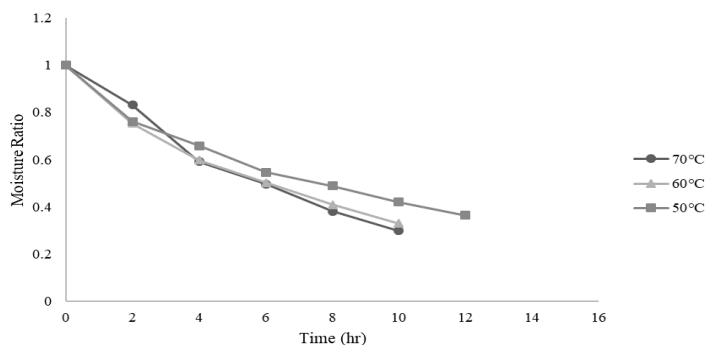

Figure 2. Effect of drying temperature 70°, 60°, and 50°C on Plum Genotype V91074

Mathematical Modelling Highest R^2, lowest Root Mean Square Value (RMSE) and Lowest Sum of Square Error (SSE) was the key criterion for the selection of the thin layer drying model which best describes the drying behavior of plum dried at three different temperatures. For all models chosen, the R^2 value was greater than 0.99, indicating a good fit (Goyal et al., 2007). Due to genotypic variation, one thin-layer drying model cannot be selected. Therefore, Modified Henderson and Pabis and Two-term exponential models were selected which can represent all genotypes studied. Similar variation for selecting the drying model can be observed in literature. As Toğrul and Pehlivan (2004)

selected Modified Henderson and Pabis to represent the drying behavior of plums, whereas, Goyal et al. (2007) selected Logarithmic model during thin layer drying of plums.

Color Analysis A significant variation in L* value of plum samples with respect to drying temperature was observed (Figure 3). Drying at elevated temperatures caused a decrease in L* value, resulting in darker color products. Similar observations for decrease in L* value with increase in drying temperature has been reported by Tarhan (2007), and Singh et al. (2013) during drying of plums and potato slices respectively. The plum's dark color can be attributed to thermal degradation of sugars (i.e Maillard Reaction) when dried at elevated temperatures. No significant difference was observed in a* and b* values with a change in temperature (Figure 3).
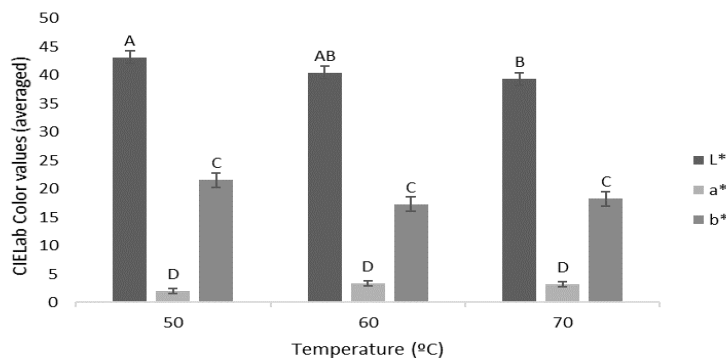

Figure 3. Change in CIELab color values with respect to temperature for all genotypes

Determination of Total Phenolic Compounds Estimation of phenolic compounds can elucidate the effect of different processing condition on physio-chemical characteristics of the sample. Total phenolic content for the fresh, freeze dried, and hot-air dried samples ranged from 3.51 to 7.61 mg GAE/ g fw, 19.75 to 54.08 mg GAE/g dw, and 13.95 to 48.57 mg GAE/g dw respectively (Table 2). Similar observation of TPC ranging between 2.11 to 8.70 mg GAE/g fw has been reported by Dowling (2014), during drying of plums. A significant increase in TPC for hot-air and freeze-dried plum was observed, as compared to fresh plums (Table 2). Similar observation for increase in TPC with respect to drying has been reported by Stacewicz-Sapuntzakis et al. (2001), and Dowling (2014), during plum drying. This trend can be attributed to increases in concentration of bio-active compounds in dried plums as compared to fresh plums. Drying creates a porous sample, which results in increase of surface area available for extraction of bio-active compounds as compared to fresh samples (Routray et al., 2014); Stacewicz-Sapuntzakis et al. (2001). Variation in TPC for each genotype with respect to temperature was observed. This trend can be due to inherent dissimilarity in their physical and chemical characteristics such as size, shape etc.

Table 2. Total phenolic content of fresh and dried Yellow European plums

	Total Phenolic Content (mg GAE/ g fresh weight)				
			Hot Air Drying		
			50°C	60°C	70°C
Genotype	Fresh	Freeze Dried			
V97261	4.25 ± 0.05[be]	27.92 ±3.13[ae]	14.62 ± 1.99[ae]	14.42 ± 0.66[ae]	14.10 ± 1.30[ae]
V94021	5.44 ± 1.47[bd]	54.08 ±3.36[ad]	26.59 ± 4.72[ad]	25.52 ± 10.10[ad]	40.98 ± 15.48[ad]
V91074	3.51 ± 0.17[bd]	47.96 ±5.91[ad]	48.31 ± 2.49[ad]	39.40 ± 3.37[ad]	43.49 ± 6.51[ad]
V98197	7.61 ± 1.17[bd]	45.71 ±3.28[ad]	48.57 ± 3.20[ad]	44.82 ± 4.08[ad]	45.89 ± 3.04[ad]
V95141	6.94 ± 0.35[be]	19.75 ±3.29[ae]	21.25 ± 2.86[ae]	13.95 ± 6.71[ae]	22.84 ± 0.78[ae]

Determination of radical scavenging activity The scavenging activity of the fresh, freeze dried, and hot air-dried plums ranged from 20.05 to 50.93%, 81.93 to 89.65%, and 35.65 to 90.58% respectively (Table 3). A significant increase in scavenging activity for hot-air dried and freeze-dried samples was observed as compared to fresh plums. Similar results for increase in scavenging activity during drying has been reported by Routray et al. (2014), and Piga et al. (2003) during drying of highbush blueberry leaves and plums respectively. This increase in scavenging activity can be attributed to increase in bio-availability of the antioxidants. Moreover, drying might have resulted in formation of new products (Millard Reaction Products (MRPs)) with higher antioxidant activity (Elizalde et al., 1992; Piga et al., 2003; Yen and Hsieh, 1995). In this study, samples dried at 50°C resulted in higher antioxidant activity as compared to ones dried at 60° and 70°C (Table 3). This variation in TPC, can be ascribed to high temperature applications for prolonged exposures, which can degrade the heat sensitive compounds. For some varieties, drying at 50°C resulted in higher scavenging activities compared to freeze drying which implies that drying at 50°C is more suitable for obtaining high DPPH inhibition activity.

Table 3. Antioxidant scavenging activity of fresh and dried Yellow European plums

	Antioxidant Scavenging Activity (%)				
			Hot Air Drying		
			50°C	60°C	70°C
Genotype	Fresh	Freeze Dried			
V97261	20.05 ± 6.50[bd]	87.57 ± 0.10[ad]	58.01 ± 8.91[ad]	45.65 ± 2.79[ad]	35.65 ± 0.55[ad]
V94021	46.76 ± 0.99[bc]	89.65 ± 0.20[ac]	78.99 ± 8.18[ac]	78.33 ± 13.14[ac]	87.16 ± 2.41[ac]
V91074	44.51 ± 3.44[bc]	81.93 ± 6.57[ac]	90.37 ± 0.10[ac]	88.27 ± 1.05[ac]	90.58 ± 0.08[ac]
V98197	46.26 ± 5.32[bc]	88.87 ± 0.26[ac]	89.92 ± 0.38[ac]	88.65 ± 1.76[ac]	89.54 ± 0.37[ac]
V95141	50.93 ± 6.90[bc]	88.20 ± 0.52[ac]	88.96 ± 1.44[ac]	68.37 ± 21.38[ac]	87.63 ± 0.98[ac]

HPLC Analysis In this study, predominant phenolic compounds that are present in plums are neo-chlorogenic acid, chlorogenic acid and ascorbic acid, which also has been reported in literature (Donovan et al., 1998; Miletić et al., 2013; Stacewicz-Sapuntzakis et al., 2001). Neo-chlorogenic acid was found to be highest amongst all the genotypes followed by ascorbic acid and chlorogenic acid. Similar observation has been reported by Piga et al. (2003), Del Caro et al. (2004) and Fang et al. (2002) during drying of plums, where they observed neo-chlorogenic acid being the highest followed by ascorbic acid and chlorogenic acid. With increase in temperature, a decrease in ascorbic acid content was observed, which can be attributed to its heat and oxidation sensitive nature (Table 4). Similar observation for decrease in ascorbic acid with increase in drying temperature has been reported by Del Caro et al. (2004), during drying of plums. Overall, hot-air drying resulted an increase in chlorogenic acid and neo-chlorogenic acid as compared to fresh plums. This trend can be ascribed to increase in bio-availability of phenolic compounds. Moreover, drying concentrates the components even with concurrent partial degradation of these constituents which results in product having higher nutrient density, phenolic and antioxidant content as compared to fresh plums (Donovan et al., 1998).

Table 4. HPLC analysis of total ascorbic acid, neo-chlorogenic acid, and chlorogenic acid content in fresh and dried YEPS

Genotype	Treatment	Ascorbic Acid (mg/g)	Neo-Chlorogenic Acid (mg/g)	Chlorogenic Acid (mg/g)
V94021	70	1.83	10.35	2.68
	60	3.50	6.22	1.93
	50	4.31	7.36	2.57
	Fresh	1.53	3.13	0.63
V91074	70	1.81	10.84	1.46
	60	1.91	7.26	0.81
	50	1.73	12.33	1.27
	Fresh	1.50	2.86	0.59

CONCLUSION The influence of dehydration on physical and chemical properties of YEPs was evaluated. YEPs were dried in hot-air at three different temperatures 50°, 60°, and 70°C. During dehydration, a falling rate period was observed. Moisture ratio data obtained in this study was fitted with 11 theoretical and semi-theoretical models to describe the drying behavior of plums. Modified Henderson & Pabis and Two-term exponential models fit most of the plum genotypes. Drying temperature had a significant effect on drying time, color and nutritional content of YEPs. Higher temperature resulted in greater colored change. Hot-air drying increased the bio-availability of the phenolic and antioxidant components as compared to fresh plums, resulting an increase in Total phenolic content and antioxidant capacity of dried plums when compared to fresh plums.

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