

Determination of drying kinetics, some physical, and antioxidant properties of papaya seeds undergoing microwave vacuum drying

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Abstract

The effects of microwave power (MP, 100–600 W) on the drying kinetics, as well as the physical and antioxidant properties of papaya seeds dried under microwave vacuum drying (MVD) were investigated. The results found that moisture diffusivity increased in the order of 10^{-8} with an increase in MP. Shrinkage increased from 1.2 to 4.9% and bulk density decreased from 3,441 to 1,091 kg/m³, respectively, while the moisture content decreased. The antioxidant contents of all of the MVD samples were higher than those of the samples dried under ambient air ventilation. An increase in MP decreased the total phenolic content from 14.69 to 10.66 mg gallic acid equivalent per gram dry weight (DW), increased both the β -carotene and the total flavonoid content in the ranges of 0.2–0.35 mg β -carotene equivalent per gram DW and 20.71 to 23.51 mg catechin equivalent per gram DW, respectively. Higher values of antioxidant activity were obtained at higher MP. The results have shown that the use of novel MVD technique in appropriate conditions can improve the overall drying efficiency and enhance the total yield of antioxidant content.

Practical Applications

Papaya seeds have great potential for use as a source of antioxidants. However, the best novel techniques for retaining high values of antioxidant compounds, and therefore recouping value from the wasted seeds, has not been widely reported on. This study has found that combined microwave vacuum drying (MVD) is a very beneficial drying method for the preservation of antioxidant content. A better antioxidant yield from dried papaya seeds was obtained using MVD at a higher MP of 600 W. Trolox equivalent antioxidant capacity of seeds dried using MVD was 27.62% higher than the samples dried using ambient air ventilation.

1 | INTRODUCTION

Papaya (*Carica papaya* L., Caricaceae) is a popular tropical fruit native to North America, and is a rich source of polyphenols, carotenoids, and vitamin C (El-Nekeety et al., 2017). In 2012, world production of

papaya was about 11.2 million metric tons (15.5% of total worldwide tropical fruit production). Papaya production in Thailand was about 212,000 tons in 2011 (FAO, 2014). The seeds of the fruit are considered waste products during processing, and are used as animal feed with no added value. However, papaya seeds contain a high amount

of total oil compounds, most of which are essential fatty acids (Malacrida, Kimura, & Jorge, 2011). Previous research has also found that the extract solution of papaya seeds is a rich source of phytochemical compounds such as phenolic acid, flavonoids, vitamin C, and possesses high antioxidant activity (Ma et al., 2017). Samaram et al. (2015) reported that papaya seed extract produced using ultrasound-assisted extraction and with oil recovery of 73% provided the highest 2,2-diphenyl-1-picrylhydrazyl (DPPH) antioxidant activity of 87.6%. Norshazila, Syed Zahir, Mustapha Suleiman, Aisyah, and Kamarul Rahim (2010) had found that the ethanolic extract of papaya seed had a total phenolic content of 8 mg gallic acid equivalent (GAE) per 100 g fresh seed weight and the seed extract scavenged 50% DPPH radicals at a concentration of 0.34 mg/mL. Zhou et al. (2011) have reported that the ethanolic extract of papaya seed contained phenolics in the amount of 1,132 mg GAE per 100 g dry weight (DW) and flavonoids of 22 mg rutin equivalents per 100 g DW. Therefore, papaya seeds have great potential for use as an antioxidant ingredient. In this case, developing a technique for preserving the high value of the antioxidant compounds represents a way to add value to the wasted seeds. Normally, fresh papaya seeds possess a high moisture content (MC) that causes easy microbial spoilage and degradation. Thus, moisture reduction methods are necessary for seed storage and use in further applications.

The drying process is an important step in the processing of medicinal plants as it could lead to either the conservation or loss of bioactive compounds and their antioxidant capacity (Onwude, Hashim, Abdan, Janius, & Chen, 2018a). Israel Sunmola, Gbenga David, Teminijesu, and Vivian (2011) found that the drying temperature (40–50°C) could increase the level of polyphenol oxidase activity in dried papaya seeds. Ironidi, Anokam, and Ndidi (2013) had reported that drying methods had significant effects on antioxidant phytochemicals such as total flavonoids, total phenols, tannins, total carotenoids, and vitamin C levels of papaya seeds, with freeze drying resulting in the highest retention of their antioxidant properties. As mentioned above, the drying process is an essential postharvest operation to maximize the shelf life of papaya seeds. Different conventional drying methods have been used to preserve agroproducts. These methods, such as conventional hot air drying, are widely used but have been reported to require longer drying times, thus resulting in lower quality products with higher nutrient degradations, while requiring increased energy consumption (Onwude, Hashim, & Chen, 2016; Onwude, Hashim, Janius, Nawi, & Abdan, 2016). Electromagnetic radiation drying methods such as microwave, infrared, and radio frequency drying are among the most highly efficient alternatives to conventional drying methods, providing high heat and mass transfer rates with low energy demands (Onwude, Hashim, Janius, et al., 2016). These novel techniques can also reduce bioproduct degradation (Bualuang, Onwude, & Pracha, 2017). Recently, these electromagnetic drying methods have been applied in combination with other conventional drying techniques in the drying of fruits and vegetables. According to Moses, Norton, Alagusundaram, and Tiwari (2014), microwave-assisted drying of food and agricultural products could reduce drying times by up to 25–90%, and energy consumption by up

to 400–800%, while increasing drying rates by up to 32–71%. In addition, the quality of the final product could also be improved when compared to the product of conventional drying methods.

Microwave vacuum drying (MVD) has been identified as one of the most significant microwave-assisted drying methods. Several studies have been carried out on the use of MVD of agricultural materials. Recently, Zielinska, Zielinska, and Markowski (2017) studied the effectiveness of microwave vacuum pretreatment before drying under a microwave vacuum. They found that the retention of both polyphenols and antioxidant activity in microwave vacuum-dried cranberries was higher than in freeze-dried cranberries. In a similar manner, Zhao et al. (2017) reported that effective moisture diffusivity increased by 127% as microwave power (MP) increased from 10 to 20 W/g. Pu and Sun (2017) observed that mango slices dried by hot air drying or MVD each exhibited individual, nonuniform drying properties. However, these studies did not consider the effect of MVD on the antioxidant capacities of agricultural products. Furthermore, a few research works have been conducted on MVD of papaya seeds. Thus, in order to achieve an effective MVD method to dry papaya seeds for commercial and industrial uses, research on factors relating to mass transfer and drying models is essential. In addition, studying the kinetics of the drying process is an important part of choosing the most suitable drying conditions and sizes of the drying equipment.

The Dincer and Dost model has been applied in estimating the mass transfer parameters of several agricultural products (Kaya, Aydin, & Dincer, 2010; Toriki-Harchegani, Ghanbarian, & Sadeghi, 2015). From the literature review, there has not been any study on the MVD of papaya seeds, nor has there been any study on the mass transfer characteristics and antioxidant capacity of papaya seeds during the MVD process. Therefore, this study aims to examine the kinetics of MVD of papaya seeds using the Dincer and Dost model at various MP levels, and also to determine the effect of MVD conditions on shrinkage percentage, bulk density, and bioactive content.

2 | MATERIALS AND METHODS

2.1 | Sample preparation

Fresh, ripe papayas (*Carica papaya* L., Caricaceae) were obtained from a local farm in Surat Thani province in Southern Thailand. Thirty pieces of papayas (50 kg) were cut into half and their seeds were collected. Seed membranes were removed, and the seeds were manually cleaned with water, then screened in order to remove excess water, and finally packed in plastic bags and stored at 4°C to await further analyses. The Association of Official Agricultural Chemists (1990) standard was used to determine the moisture content (MC) of the seeds, and calculated using Equation (1). Approximately 10 g of the sample was dried in a hot air oven at 105°C for 24 hr and then kept in a desiccator until the grain temperature reached that of the ambient air. The accuracy of the initial and final weights of the samples was determined by using a digital balance (Mettler Toledo- PL1502-S, with 0.01 g accuracy). The experiments were carried out in triplicate and then reported in terms of the average value of $MC \pm SD$.

$$MC = \frac{(w-d)}{d}, \tag{1}$$

$$MR = \frac{M_t - M_e}{M_0 - M_e}, \tag{2}$$

where MC is the moisture content (g H₂O/g DW), and *w* and *d* are the weights of the moist and dry seeds, respectively.

2.2 | Microwave vacuum drying

The microwave vacuum dryer consisted of a domestic microwave following the design of Monteiro, Link, Tribuzi, Carciofi, and Laurindo (2018), with some modifications, was used for these drying experiments. The drying system comprised of a microwave oven (MS23F301E model, Samsung, Thailand) with dimensions of 489 × 275 × 392 mm, a maximum MP output of 800 W, and a microwave frequency of 2,450 MHz. A vacuum pump was connected to the vacuum chamber. A picture of the microwave vacuum dryer is shown in Figure 1. This domestic microwave oven was modified into a microwave vacuum dryer. The dryer contains of a vacuum chamber (18 cm diameter and 12 cm high) placed into the oven cavity with dimensions of 330 mm (*W*) × 211 mm (*H*) × 324 mm (*D*). The vacuum chamber was connected to a vacuum pump (New IM-TECH, model IM125D, Thailand) with a pumping rate of 0.108 m³/min and a power supply of 220–240 V 50 Hz. A pressure gauge was used for measuring the pressure inside the chamber. The water vapor from the dryer was removed via the vacuum pump line and was adsorbed into a column of silica gel. Sixty grams of the cleaned seeds were taken from the refrigerator and placed in ambient air temperature until the grain temperature was the same as that of its surroundings. The drying experiments were carried out in the microwave vacuum dryer at 100, 300, 450, and 600 W. Sixty grams of fresh seeds were placed into a vacuum chamber put into the oven compartment of the microwave vacuum dryer. The pressure inside the chamber was reduced to 100 mbar (around 10 s) before turning on the microwave oven and remained stable until the desired drying time was reached. During the drying process, the evolution of the seed mass was measured at 2-min intervals until reaching the required MC. Grain surface temperature was measured by an infrared thermometer (RS Pro Model RS1327 brand Amprobe). After drying, the dried samples were kept in a plastic bag at 4°C.

The moisture ratio (MR) during the drying process was calculated using the following equation (Bualuang et al., 2017):

where MR is the dimensionless moisture ratio, *M_t* is the MC at any given drying time (g H₂O/g DW), *M₀* is the initial MC (g H₂O/g DW), and *M_e* is the equilibrium MC (g H₂O/g DW). Due to the small value of the equilibrium MC, the MR was simplified to Equation (3) (Nadi & Abdanan, 2017).

$$MR = \frac{M_t}{M_0}. \tag{3}$$

2.3 | Mass transfer characteristics

The mass transfer characteristics of agricultural products can be described using the Dincer and Dost model (Onwude et al., 2018a). This mass transfer model provides a one-dimensional diffusion solution for various product shapes. Several assumptions were made for the effective application of this model, including constant thermophysical properties of the drying material and drying medium, one-dimensional moisture diffusivity in the radial direction of the product (sphere), and negligible effects of heat transfer on mass transfer. Equations 4–7 depict the moisture diffusivity equations of papaya seeds given the above assumptions:

$$\left(\frac{1}{r^2}\right) \left(\frac{\partial}{\partial r}\right) \left(r^2 \frac{\partial M}{\partial r}\right) = \left(\frac{1}{D}\right) \left(\frac{\partial M}{\partial t}\right), \tag{4}$$

$$M(r,t) = M_0, t = 0, 0 \leq r \leq R, \tag{5}$$

$$\frac{\partial M}{\partial r}(r,t) = 0, t > 0, r = 0, \tag{6}$$

$$-D \left(\frac{\partial M}{\partial r}\right)(r,t) = k_m(M_s - M_e), t > 0, r = R, \tag{7}$$

where *r* is the distance from the center (m), *R* is the sample radius (m), *k_m* is the mass transfer coefficient (m/s), *t* is drying time (s), *D* is the moisture diffusivity (m²/s), *M* is the MC, *M_s* is the MC of the sample surface (g H₂O/g DW), and *M_e* is the equilibrium MC of the sample.

To determine the MR, moisture diffusivity, and mass transfer coefficient, the Dincer and Dost equation was applied. The curve fitting method of plotting MR against drying time (*t*) was used to

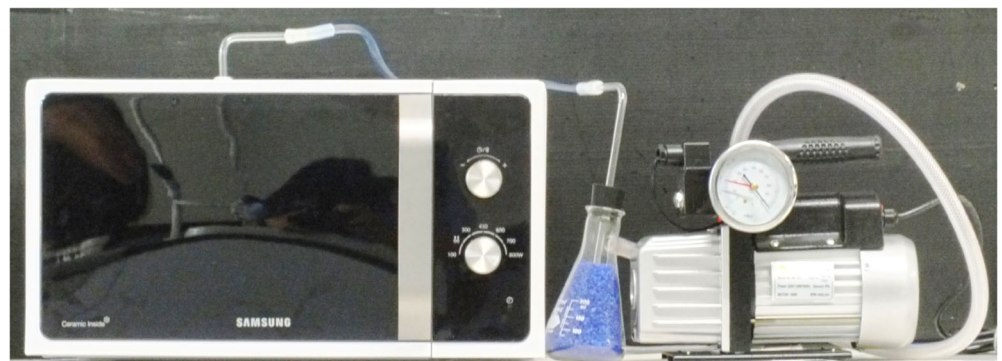


FIGURE 1 A microwave vacuum dryer

estimate the drying coefficient (S) and lag factor (G) as expressed in Equation (8).

$$MR = G \exp(-S \cdot t). \quad (8)$$

The Biot number (Bi) was calculated using Equation (9):

$$G = \exp\left(\frac{0.7599 Bi}{2.1 + Bi}\right). \quad (9)$$

The value of the first root of the transcendental characteristic (μ_1) was determined using Equation (10):

$$\mu_1 = (1.1223 \times \ln(4.9 Bi + 1))^{1/4}, \text{ for } 0.1 < Bi < 100. \quad (10)$$

The moisture diffusivity (D) was determined using Equation (11):

$$D = \frac{S \times R^2}{\mu_1^2}. \quad (11)$$

The mass transfer coefficient (k_m) was estimated as follows:

$$k_m = \frac{D \times Bi}{R}. \quad (12)$$

To validate the results obtained from the model, the MR of the papaya seeds was calculated using Equation (4). Assuming the stated initial and boundary conditions, the MR of the papaya seeds at any given point is as follows (McMinn, Khraisheh, & Magee, 2003):

$$MR = \sum_{n=1}^{\infty} A_n B_n. \quad (13)$$

A_n and B_n are defined as Equation (14):

$$\begin{cases} A_n = \frac{2 \sin \mu_n}{\mu_n + \sin \mu_n \cos \mu_n}, & 0.1 < Bi < 100, \\ A_n = \frac{2(-1)^{n+1}}{\mu_n}, & Bi > 100, \end{cases} \quad (14)$$

$$B_n = \exp(-m_n^2 F_0) \text{ for } 0.1 < Bi < 100 \text{ and } Bi > 100, \quad (15)$$

where m_n is the n th root of the transcendental characteristic equation and F_0 is the Fourier number calculated using Equation (16).

$$F_0 = \frac{Dt}{R^2}. \quad (16)$$

Ignoring small values of the Fourier number, and applying only the first term of the series, Equation (13) can be simplified to Equation (17):

$$MR \gg A_1 B_1, \quad (17)$$

where $A_1 = G$,

$$B_1 = \exp(-m_1^2 F_0). \quad (18)$$

The MR from Equation (17) was fitted to the experimental data to validate the Dincer and Dost model.

The correlation coefficient (R^2) and root mean square error (RMSE) were used in evaluating the ability of the model to fit the experimental data. These parameters are defined as follows:

$$R^2 = 1 - \frac{\sum_{i=1}^n (MR_{\text{exp},i} - MR_{\text{pre},i})^2}{\sum_{i=1}^n (MR_{\text{exp},i} - \overline{MR_{\text{exp}}})^2}, \quad (19)$$

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^N (MR_{\text{pre},i} - MR_{\text{exp},i})^2 \right]^{1/2}, \quad (20)$$

where MR_{exp} is the experimental data, MR_{pre} is the data predicted by the model, $\overline{MR_{\text{exp}}}$ is the mean of experimental data, and N is the number of data points.

2.4 | Shrinkage percentage (% shrinkage)

The shrinkage evolution of agricultural food materials during the drying process is a common phenomenon directly affecting the final product's overall quality (Onwude et al., 2017; Onwude, Hashim, Janius et al., 2016). Drying method, drying condition, and MC affect shrinkage and the resultant bulk density of the seeds (Koua, Koffi, & Gbaha, 2017; Serowik et al., 2017). During the drying process, the moisture was removed from the sample, resulting in a change in the structural composition of the seed. In this study, shrinkage percentage was expressed as a ratio of the decrease of dried seed sphericity (ϕ) to seed sphericity of the moist sample. One hundred seeds randomly selected from the sample were measured using a Vernier caliper (Mototaro, Thailand) with an accuracy of 0.01 mm in x , y , and z geometric dimensions as shown in Figure 2. The sphericity was defined as a ratio of the volume of the sample, which is assumed to be equal to the volume of the triaxial ellipsoid having equivalent diameters, to the volume of the circumscribed sphere. The sphericity of each seed was calculated following Equation (21) (Mohsenin, 1970):

$$\phi = \frac{(xyz)^{1/3}}{x}. \quad (21)$$

Shrinkage percentage was determined using Equation (22) (Tapaneyasin, Devahastin, & Tansakul, 2005).

$$\% \text{ Shrinkage} = \frac{\phi_{\text{initial}} - \phi_{\text{final}}}{\phi_{\text{initial}}} \times 100, \quad (22)$$

where ϕ_{initial} and ϕ_{final} are the sphericities of the papaya seeds at the beginning and at the end of the drying experiment, respectively.

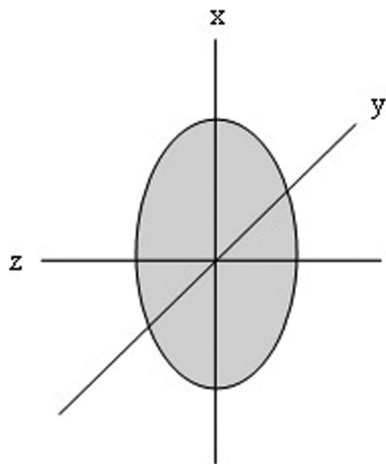


FIGURE 2 Geometric dimensions x , y , and z of seed measurement

2.5 | Bulk density (ρ)

Bulk density is defined as the DW of the particles divided by the overall volume of the particles. Bulk density considers both solid material and pore space. The effect of drying on bulk density can be explained by the change in product volume due to shrinkage. The modifications of material structures including bulk density can significantly influence the process performance, for example, drying rate and both mass and heat transfers (Qiu et al., 2015). Bulk density is expressed as the ratio of sample mass to the total volume, including volume of the voids between grains. Bulk density was determined using the samples, which had five different MCs. Grains with the same MC were placed in 10 mL cylinders until full. After that, the cylinders were weighed with a digital balance (PL1502-S model manufactured by Mettler Toledo with 0.01 g accuracy, Thailand). The experiment was done 10 times at each MC level. The bulk density was calculated using Equation (23):

$$\rho = \frac{m}{V}, \quad (23)$$

where ρ is the bulk density (kg/m^3), m is the mass of the sample (kg), and V is the whole volume of sample (m^3).

2.6 | Antioxidant analysis

2.6.1 | Sample extraction

Dried papaya seeds were ground into fine particles using a blender. A methanolic extract of the sample was prepared following the method of Chan, Lim, and Chew (2007) with some modifications. A 10 g sample of the ground particles was extracted in duplicate using 50 mL of methanol for 6 hr at room temperature and stirred thoroughly with a magnetic stirrer. Then the slurry was filtered through Whatman filter paper. The residual particles were then extracted with the same volume of methanol. The filtrate was collected and evaporated using a vacuum evaporator (Rotavapor R-215 Buchi, Zurich, Switzerland) at 40°C. After that, the concentrated solutions were dissolved with

25 mL of methanol before being stored in a refrigerator at 4°C to await further analysis.

2.6.2 | Determination of total phenolic content

Total phenolic content (TPC) was analyzed following the Folin-Ciocalteu method using gallic acid as a standard solution (Singleton & Rossi, 1965). In short, a diluted sample solution that had absorbance in the ranges of 0.100–0.800 was prepared. The sample extract solution of 0.5 mL, standard gallic acid solution, was pipetted to a test tube; 2.5 mL of distilled water and 0.5 mL of 10% vol/vol Folin-Ciocalteu reagent were added to the test tube and vortexed using a mixer. After 8 min of the reaction, 1.5 mL of 20% wt/vol sodium carbonate solution (Na_2CO_3) was added and mixed thoroughly. After incubation at ambient temperature for 2 hr, the absorbance of the mixture was measured at 722 nm using UV visible spectrophotometry. The gallic acid solution of 0–80 mg/L was used for the calibration curve. The phenolic content was calculated and expressed as milligram of GAEs per gram of dried papaya seed (mg GAE/g DW). All of the experiments were conducted in triplicate.

2.6.3 | Determination of TFC

The AlCl_3 colorimetric method as explained by Anna, Dae-Ok, Sang-Jin, Sung, and Ock (2010) was used in determining the TFC of the papaya seeds. Appropriately diluted extracts were prepared to obtain the absorbance in the range of 0.100–0.800. Diluted sample of 3 mL or catechin solution, 9.4 mL of distilled water, and 0.6 mL of 5% wt/vol sodium nitrite (NaNO_2) were combined and mixed thoroughly. The resultant mixed solution was then left for 5 min and 0.6 mL of 10% wt/vol aluminum chloride (AlCl_3) and 3 mL of 1 M of sodium hydroxide (NaOH) were added to the mixture. The absorbance was measured at 487 nm after 15 min of reaction time. Catechin solution (0–350 mg/L) was used for the standard curve. Flavonoid content was calculated and expressed in terms of mg of catechin equivalents per gram of DW (mg CAE/g DW). All experiments were carried out in triplicate.

2.6.4 | Determination of β -carotene content

β -Carotene content (BCC) was analyzed based on the method derived by Kriengsak, Unaroj, Kevin, Luis, and David (2006) with some modifications. Suitable diluted solution of 2 mL (0.100 to 0.800) was pipetted. The absorbance of the extracted solution was measured at 450 nm, and a concentrated β -carotene solution (0–160 ppm) was used as the standard solution in each analysis. The BCC was presented as milligram of β -carotene equivalents per gram DW (mg BCE/g DW). All determinations were done in triplicate.

2.6.5 | Antioxidant degradation rate

Degradation of antioxidant compounds was assumed to be a first-order reaction following Equation (24):

$$\ln\left(\frac{C}{C_0}\right) = -kt, \quad (24)$$

where C and C_0 are the concentrations of antioxidant compounds in the sample with and without drying at any given drying time (t), respectively, and where k is the degradation rate constant.

2.6.6 | Antioxidant activity

2,2'-azino-bis (3-ethylbenzthiazoline-6-sulphonic acid (ABTS⁺)) is a stable cation radical which was prepared via the reaction between ABTS and potassium persulfate using 7.5 mM ABTS and 2.45 mM potassium persulfate, and incubated at room temperature in the dark for 12–16 hr before use (Rajurkar & Hande, 2011). The generated ABTS⁺ radical was blue-green in color. Then, an ABTS⁺ diluted solution with an absorbance of 746.5 nm in the range of 0.800–0.100 was prepared. The extract solution of 0.1 mL was mixed with 3.9 mL of the ABTS⁺ solution and then shaken thoroughly. The ABTS radical was then bleached by receiving the hydrogen atom or electron from antioxidant compounds contained in the extract solution. The absorbance of the mixture was measured at 746.5 nm. Trolox standard solutions with concentrations varying from 0 to 180 mg/L were prepared for the standard curve. Scavenging capacity of ABTS radical cation was measured and expressed in the terms of % radical scavenging activity:

$$\% \text{radical scavenging activity} = \left[\left(\frac{A_0 - A_s}{A_0} \right) \right] \times 100, \quad (25)$$

where A_s and A_0 are the ABTS solution absorbance with and without the tested sample, respectively.

Antioxidant activity of the extract solution was expressed as IC50 and Trolox equivalent antioxidant capacity (TEAC). IC50 of the sample was evaluated from the curve of radical scavenging percentage against the concentration of extract solution. TEAC was evaluated in milligram Trolox equivalent per gram dried weight (mg TE/g DW) as expressed in Equation (26). The IC50 of Trolox is 104.52 mg/mL.

$$\text{TEAC} = \frac{\text{IC50}_{\text{Trolox}}}{\text{IC50}_{\text{sample}}}. \quad (26)$$

3 | RESULTS AND DISCUSSION

3.1 | Lag factor, drying coefficient, Biot number, and dimensionless root of transcendental characteristics

The papaya seeds, with an initial MC of 2.80 (g H₂O/g DW), were dehydrated using a microwave vacuum dryer until reaching an average final MC of 0.11 (g H₂O/g DW), a level which is safe for storage. To describe the moisture transfer kinetics of MVD, the mathematical models were useful in predicting the mass transfer mechanism, which is necessary for the appropriate process parameter selection and for improving drying equipment design. Therefore, drying kinetics

TABLE 1 Values for drying parameter, antioxidant degradation rate, and TPC, BCC, TFC, and TEAC of papaya seed dried under MVD

MP (W)	Dincer and Dost model				Drying parameter				Degradation rate constant				Antioxidant content					
	G	S × 10 ³ (/s)	R ²	RMSE	Bi	μ ₁	D × 10 ⁸ (m ² /s)	k _m × 10 ⁶ (m/s)	k × 10 ³ (/s)	TPC (mg GAE/g DW)	R ²	BCC (mg BCE/g DW)	k × 10 ³ (/s)	TFC (k × 10 ³ (/s))	R ²	TPC (mg GAE/g DW)	BCC (mg BCE/g DW)	TFC (mg CAE/g DW)
100	1.04	0.53	.947	0.063	0.21	0.47	1.07	1.04	0.47	.849	0.67	.930	0.43	.864	13.292 ^c	0.046 ^e	23.640 ^d	0.1181 ^f
300	1.07	1.38	.946	0.077	0.49	0.61	1.69	3.92	1.31	.926	1.77	.988	1.25	.956	14.694 ^b	0.059 ^d	25.364 ^c	0.1217 ^e
450	1.08	2.13	.950	0.082	0.56	0.63	2.40	6.25	2.22	.991	2.34	.984	1.69	.999	11.152 ^d	0.065 ^c	26.091 ^b	0.1303 ^c
600	1.07	2.61	.956	0.081	0.43	0.58	3.53	7.07	2.25	.952	2.53	.932	1.95	.989	10.661 ^e	0.069 ^b	25.981 ^b	0.1502 ^b
Control	-	-	-	-	-	-	-	-	-	-	-	-	-	-	10.039 ^f	0.060 ^d	19.674 ^e	0.1281 ^d
Fresh	-	-	-	-	-	-	-	-	-	-	-	-	-	-	64.441 ^a	0.238 ^a	83.022 ^a	0.4932 ^a

Note: MP is the microwave power, G is the lag factor, S is the drying coefficient, R² is the correlation coefficient, RMSE is root mean square error, Bi is Biot number, μ₁ is dimensionless root of transcendental characteristic; D is the moisture diffusivity and k_m is mass transfer coefficient (the values of D and k_m are estimated assuming that the sample radius is 0.002993 m). k is the degradation rate constant; TPC, BCC, TFC, and TEAC are total phenolic content, β-carotene content, and Trolox equivalent antioxidant capacity, respectively; control is papaya seed dried under ambient air ventilating. Different letters (a–f) in the same column of antioxidant content mean significant difference among different microwave power drying condition (p < .05).

parameters were evaluated using the Dincer and Dost model. Table 1 illustrates the lag factor (G) and drying coefficient (S) which were estimated using a nonlinear regression curve fitting method derived from the plotting of the MR versus drying time according to Equation (8). High values of R^2 ($R^2 > .95$) and low RMSE ($RMSE < 0.08$) were observed, implying that the Dincer and Dost model fit the experimental data adequately.

The effect of MP on drying parameters is shown in Table 1. As expected, during MVD, the change in MP has a strong effect on the drying process. The drying coefficient increased with increasing MP levels. This was because of rapid heat and mass transfer at higher MP levels. The obtained lag factor in this study was in the range of 1.036–1.079, signifying that both internal and external resistance controlled the mass transfer of sample grains during the drying process. These lag factor values are similar to previous results for whole lemon (Torki-Harchegani et al., 2015).

The estimated Bi number varied from 0.208 to 0.555, demonstrating the existence of internal and external resistance to mass transfer. In addition, the Bi number values depended on MP levels. Similar results were observed by Beigi (2016) for sliced apple, Onwude, Hashim, Abdan, Janius, and Chen (2018b) for sweet potato, and Torki-Harchegani et al. (2015) for whole lemon.

3.2 | Drying curve, moisture diffusivity, and mass transfer coefficient of papaya seed

The Dincer and Dost model Equation (17) was used to predict the MR of papaya seeds dried by MVD. The effects of MP (at 100, 300, 450, and 600 W) on moisture removal were determined. The moisture transfer shown in terms of MR evolution during the drying period is illustrated in Figure 3. The results showed that the Dincer

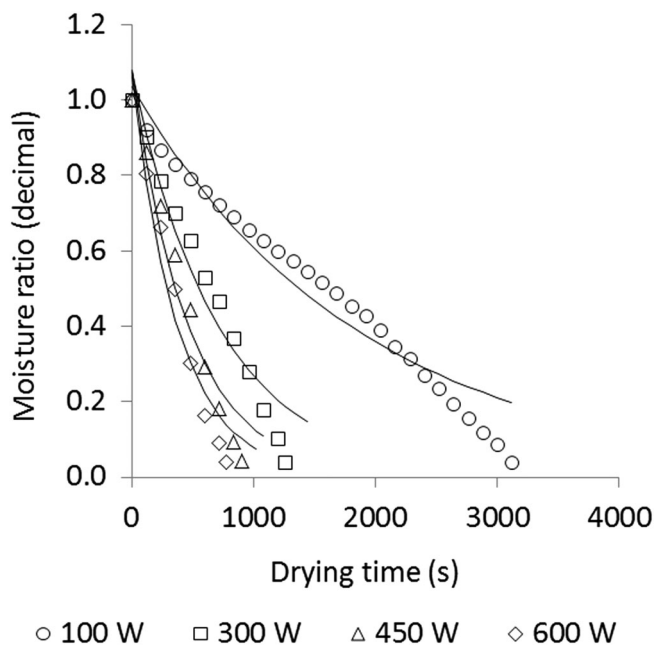


FIGURE 3 Moisture ratio of papaya seed dried under microwave vacuum drying at different microwave power

and Dost model resulted in accurate predictions of the experimental data with high R^2 and low RMSE values. It was also observed that MP has a strong effect on drying time. As expected, the MC decreased faster at higher MP levels in all drying experiments due to the increase in temperature. At the same MR level, the drying time decreased with increased MP level indicating that more heat was generated within the sample. Reducing the MC of the seeds from 2.80 to 0.11 g H_2O/g DW took 13–52 min of drying time. At the highest MP, drying time could be reduced by up to 75.0% as compared with drying at the lowest MP. Furthermore, at high MP levels, the drying rate exhibited the falling rate period instead of the constant rate phase as often observed for conventional drying (Ambros, Foerst, & Kulozika, 2018), indicating that diffusion is the main mechanism of moisture removal (Demiray & Tulek, 2017). This result is similar to that of Monteiro et al. (2018).

The moisture diffusivity (D) of papaya seeds during MVD as estimated by the Dincer and Dost model using Equation (11) is shown in Table 1. The moisture diffusivity was found to increase in the range of 1.07×10^{-8} to 3.53×10^{-8} m^2/s with an increase in MP from 100 to 600 W. The D values were increased by up to 229.9% at 600 W when compared with 100 W, while the drying time decreased by 75.0%. This is because higher MP creates increased driving force for heat and mass transfer within the drying material. The MC rapidly decreased at higher MP thereby increasing vapor penetrability. As drying continues, the MC of the sample absorbs microwave energy while the pressure inside the grains is raised. The moisture pressure opens the pore structure allowing for increased moisture diffusion. Similar results were found in the literature for spherical onion (Demiray & Tulek, 2017) and for spherical germinated maize grain (Bualuang et al., 2017). In addition, the MP had a significant effect on moisture diffusion. The results have shown that the D level increased with increased MP in an exponential relationship with good accuracy ($R^2 > .999$).

The values of the moisture transfer coefficient (k_m) increased linearly in the range of 1.04×10^{-6} to 7.07×10^{-6} m/s with both an increase in MP and R^2 close to .970. This is consistent with other studies such as whole lemon (Torki-Harchegani et al., 2015) and sliced apple (Beigi, 2016).

Grain temperature evaluation during the drying process was illustrated in Figure 4. The pressure inside the chamber for all MP tests was maintained at 100 mbar. Grain temperature was higher than the boiling point of water ($45.6^\circ C$ at 100 mbar) for 300–600 MP levels, at the beginning of the drying process because of the ebullioscopic effect (Ambros et al., 2018). Grain temperature rose rapidly due to increased drying time except for 100 W. At 100 W MP, grain temperature seems to be constant and lower than the theoretical boiling point at the first stage of drying. After 2,400 s of drying, temperature rose to a higher boiling level due to a high energy of absorption. From 300 to 600 W power input, the grain temperature increased rapidly past the boiling point, as surface moisture decreased. Therefore, the MP input level affected the grain temperature due to the large microwave energy absorption by the grain during the initial drying stage, which differed from conventional drying processes (Ambros et al., 2018).

3.3 | Shrinkage percentage (percent shrinkage) and bulk density (ρ)

Figure 5a illustrates the shrinkage development at different MP values. The results show that the shrinkage curve and MC have a strong linear correlation. This is because when moisture is removed from a texture, a pressure difference is generated between the inside and outside of the texture, thus leading to shrinkage. As shown in the figure, shrinkage rapidly increased with decreasing MC in papaya seeds. In addition, an increase in MP level from 100 to 600 W resulted in a 0.79% increase in shrinkage at the final MC of 0.11 (g H₂O/g DW). This result shows that deformation of papaya seeds during MVD increases with the moisture removal rate, leading to increased stress on the cellular material caused by contraction.

Overall, shrinkage percentage of MVD was in the range of 1.2–4.9%. Shrinkage at higher MP (450 W and 600 W) was obviously

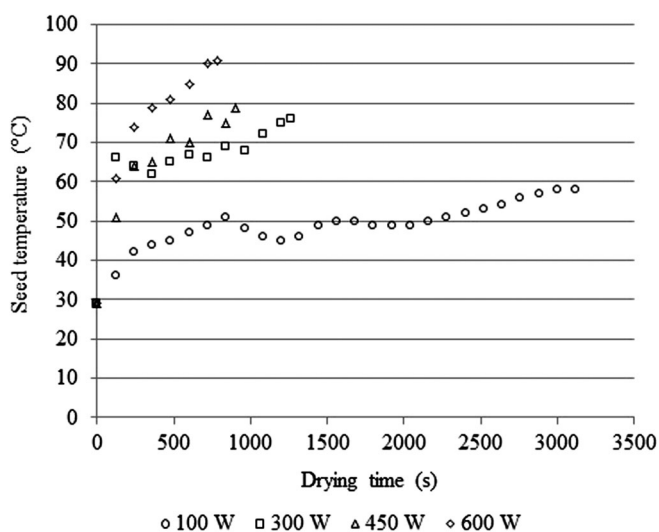


FIGURE 4 Grain temperature during drying process at different microwave power

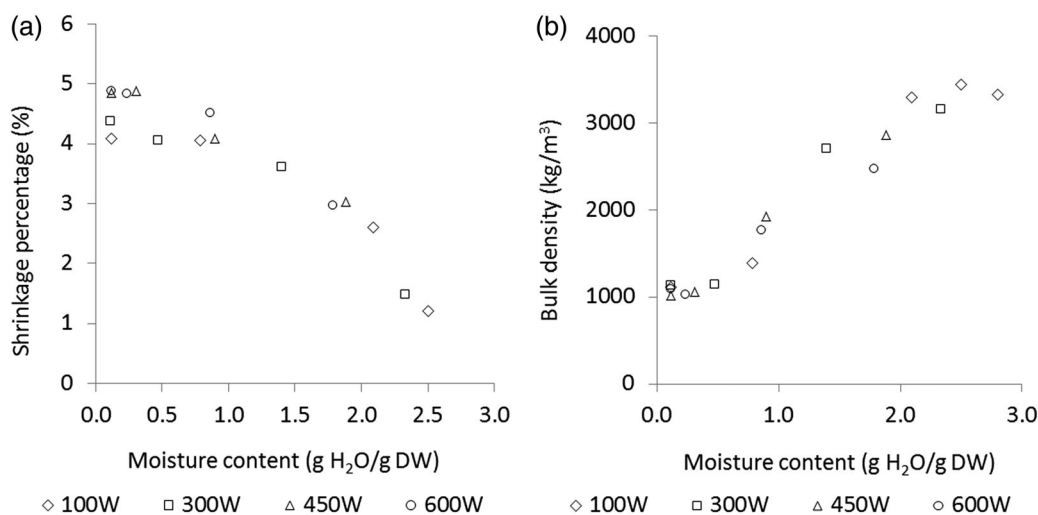


FIGURE 5 Shrinkage percentage (a) and bulk density (b) of papaya seed at different moisture content

higher than at lower MP according to previous work (Nahimana & Zhang, 2011). The authors also reported that the increase of the radial shrinkage values of sliced carrot ranged from 36.2 to 46.1% for MVD as MP level increased from 1.3 to 2.5 W/g of the sample.

Figure 5b shows the bulk density of the papaya seed samples at different MCs during the drying process. The graph clearly indicates that the bulk density is significantly affected by the MC. Bulk density decreased linearly from 3,441 to 1,091 kg/m³ as MC decreased from 2.80 to 0.11 g H₂O/g DW. Final bulk density was found to be unaffected by dehydration energy. No clear behavioral effect of MP on the bulk density was observed. A similar phenomenon was also described by Serowik et al. (2017) for other fruits and vegetables.

3.4 | Effect of MVD on antioxidant compounds

Figure 6a–c shows the average values of TPC, BCC, and TFC, respectively, for MVD dried papaya seeds at 100, 300, 450, and 600 W. The results show that the antioxidant retention of the sample rapidly decreased during the initial drying period and then the retention was relatively constant. This is because some water-soluble molecules may greatly stimulate the decay process at high moisture levels during the initial drying period (Goula & Adamopoulos, 2006). Moreover, the rapid thermal degradation of antioxidant compounds during the initial drying period may be due in part to high levels of heat conversion resulting from high microwave energy, leading to increased sample temperatures. A temperature plateau was attained when the absorbed microwave energy was balanced by the energy losses associated with moisture evaporation and surface convective cooling. As the drying process proceeded beyond the constant temperature period, the rate of moisture loss was significantly reduced. The absorbed microwave energy decreased in this stage which led to a decrease in the rate of antioxidant degradation (Lu, Tang, & Ran, 1999). During the drying of agricultural products, the degradation of antioxidant compounds such as polyphenolics often occur, resulting in a decrease in antioxidant activity (Lech, Figiel, Michalska, Wojdyło, & Nowicka, 2018).

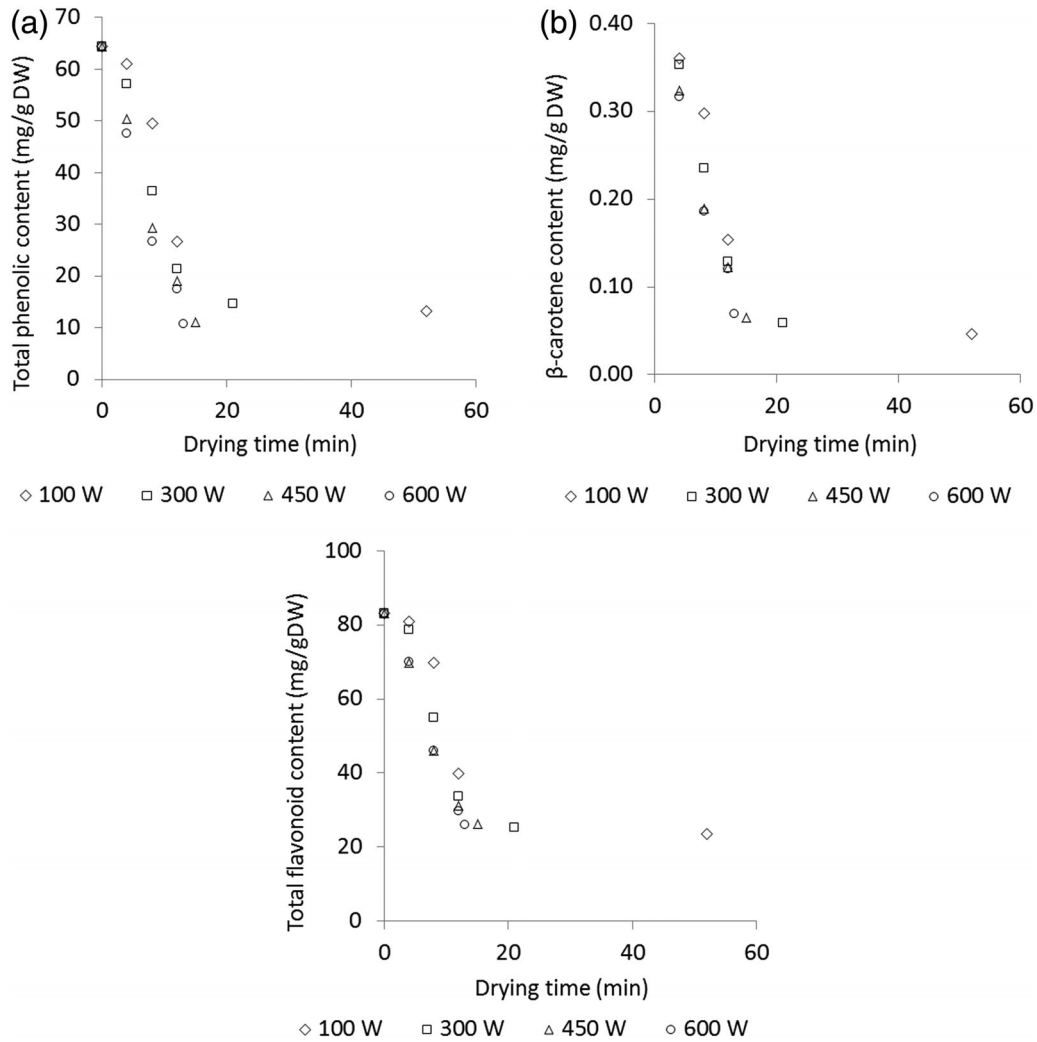


FIGURE 6 (a) Phenolic content, (b) β-carotene, and (c) flavonoid content of papaya seed under microwave vacuum drying at different microwave energy

The degradation kinetics of TPC, BCC, and TFC were described using the first-order reaction equation at each MP level with a high degree of correlation ($R^2 > .85$). Table 1 illustrates the degradation rate constant (k) with high relative correlation for antioxidant first-order degradation. The results show that degradation increased rapidly at high MP. This is because of the relatively large MP absorbed by the product at a higher microwave energy/mass ratio, which led to increased sample temperature (Lu et al., 1999).

At the final drying stage, a MC of 0.11 g H₂O/g DW and approximately 16.54% retention of TPC was observed at 600 W (drying time was 780 s), while at 100 W, the retention reached 20.63% (Table 1). However, it was also found that the highest retention level of TPC was 22.80% when drying at 300 W. This may be because of the combined effect of both shorter drying time and a mild thermal application of 300 W, which helped to retain phenolic compounds otherwise lost during the drying process. In contrast, Kammoun Bejar, Kechaou, and Boudhrioua Mihoubi (2011) found that drying with microwaves at a power of 450 W significantly improved the extraction of TPC from citrus peel. According to these authors, at this MP, the structure of the

fiber matrix become larger and looser and thereafter helped facilitate extraction with solvent. The highest contents of polyphenols in microwave-dried sage plants could be explained by the fact that the intense heat generated from the microwaves creates both high vapor pressure and temperature inside plant tissue, resulting in the decomposition of plant cell wall polymers. Consequently, in certain cases, cell wall phenolics or bond phenolics could be released, thus causing more phenolics to be extracted (Inchuen, Narkrugsa, & Pornchaloempong, 2010). As for microwave drying, literature review found widely variable results including both decrease and increase of TPC depending on the plant material. Indeed, Sathishkumar, Lakshmi, and Annamalai (2009) reported a sharp decrease in the TPC of *Enicostemma littorale* as processed by microwave drying. Inchuen et al. (2010) have reported that increasing MP from 180 to 540 W resulted in an increase of the TPC of Thai red curry powder. Kammoun Bejar et al. (2011) found that the drying method deeply affected qualitative phenolic composition by increasing contents of some compounds but decreasing those of others, as well as by the appearance of some other phenolics such as transcinnamic acid and 4',5,7-trihydroxyflavone while other phenolic compounds were lost.

In the drying treatment of plants, according to Mueller-Harvey (2001), heating not only deactivates enzymes, but also degrades phytochemicals. Some phenolic compounds decompose rapidly in direct sunlight or if dried at elevated temperatures.

The effect of MP on BCC was also shown in Table 1. The results show that an increase in MP with shorter drying period reduced BCC degradation. The highest retention of β -carotene (29.93%) was found when drying at 600 W and the lowest retention level of 20.09% was found when drying at 100 W. In the presence of catalysts, because carotenoids are light, heat, and oxygen sensitive, the conjugate double bonds react with oxygen and other radicals to form carotenoid oxidation products and/or other derived products. The MVD environment drives out oxygen and causes water to evaporate at low temperatures as described by Nahimana and Zhang (2011). Hiranvarachat, Suvarnakuta, and Devahastin (2008) have reported that drying conditions and drying techniques significantly affected β -carotene degradation. Suvarnakuta, Devahastin, and Mujumdar (2005) also found that drying in an oxygen-free environment (vacuum condition) or with short air exposure time led to a reduction of β -carotene degradation. Therefore, the best conditions for retaining β -carotene are an oxygen-free environment where MP is 600 W, thus requiring less exposure time.

A similar trend has been observed in flavonoid levels. The thermal heating and drying time also have significant effects on TFC. Less retention of TFC (24.95%) was shown at 100 W while the highest retention of TFC was 28.32% during MVD at 600 W. In addition, drying at 600 W MP showed a higher level of TPC, BCC, and TFC with 5.84%, 12.79%, and 16.33%, respectively, when compared with the control sample dehydrated by ambient air ventilation. The lower levels found in the control sample were due to the fact that the hydroxyl group of phenolics, flavonoids, and other antioxidant compounds were easily oxidized in an oxygenated environment.

3.5 | Effect of MP on ABTS scavenging activity in papaya seeds

The TEAC of the extracts obtained from papaya seeds dried at different MP was illustrated in Figure 7. At the conclusion of the drying stage (when MC of the sample reached 0.11 g H₂O/g DW) the TEAC increased in the range of 0.122–0.150 mg TE/g DW with an increase in MP. Moreover, it was observed that TEAC of seeds dried by MVD was 27.616% higher than the control sample dried using ambient air ventilation. This was due to the short drying period which could have preserved antioxidant compounds. Thermal drying significantly influenced ($p < .05$) the antioxidant potential of the extracts. Thus, the increase in MP from 100 to 600 W led to a significant increase in TEAC ($p < .05$). Also, the highest TFC, BCC, and TEAC values corresponded to papaya seeds dried at the highest MP. MVD was carried out at safe temperatures (below 90°C) for the grain at all MP inputs (Lech et al., 2018). An increase in the antioxidant activity of MVD plants with increased MP has been reported by several authors such as Dong et al. (2018) in the methanolic extract of green coffee beans, Michalska, Wojdyło, Lech, Łysiak, and Figiel (2017) in the methanolic extract of black-currant powder, and Figiel (2010) in the extract of beetroot. MVD drying

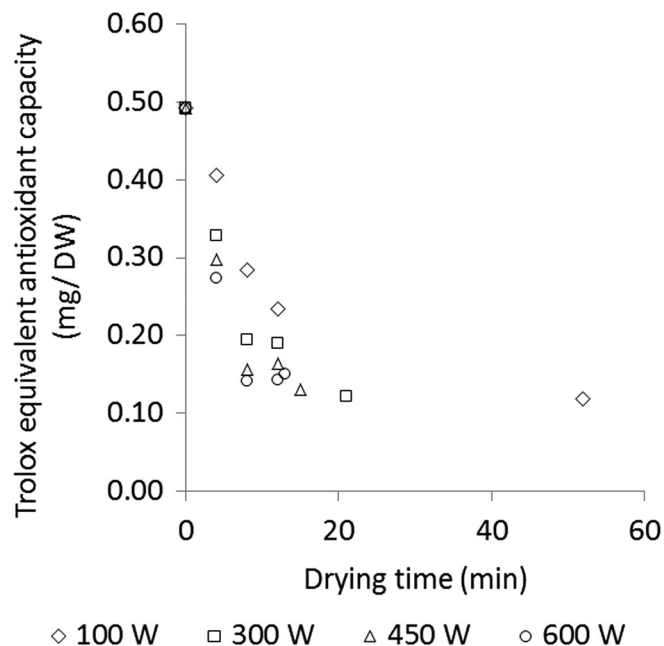


FIGURE 7 Antioxidant activity of papaya seed at different microwave power of microwave vacuum drying

seems to enhance TFC, BCC, and TEAC ABTS. Most of these parameters increased with an increase of MP from 100 to 800 W.

According to Niamnuy, Nachaisin, Jamradloedluk, and Devahastin (2010), drying at higher temperatures may be the cause of changes in the sample's structure. Therefore, during the grinding of samples, phenolic, β -carotene, flavonoid compounds, and other intracellular compounds are released. Moreover, some nutrient molecules can be transformed into compounds that have higher antioxidant capacities. This includes the transformation of malonyl- and acetyl-glycoside isoflavones compounds found in soybeans dried at higher thermal intensities into other compounds possessing higher antioxidant power, thus causing increases in DPPH and ferric ion reducing activity power (FRAP). Ahmad-Qasem, Barrajón-Catalán, Micol, Mulet, and García-Pérez (2013) have reported that increased TEAC of olive leaves was due to increased drying temperatures. The same trend was also observed for coconut juice mixed with coconut flesh. The antioxidant potential, DPPH, FRAP, and superoxide anion radical scavenging activity were significantly increased at higher thermal processing conditions ($p < .05$) (Juntachote, 2013). López et al. (2010) found that hot air drying at high temperatures caused degradation of TPC in blueberries, simultaneously raising antioxidant activity.

4 | CONCLUSIONS

This research investigated the MVD of papaya seeds at different MPs. The effect of MVD conditions on shrinkage, bulk density, and bioactive content of the papaya seeds was also examined. This MVD method has shown that the drying conditions had significant effects on the drying kinetics, physical properties, and phytochemical levels of papaya seeds. The Dincer and Dost model was able to adequately describe the

mechanism of mass transfer. The moisture diffusivity for papaya seeds varied from 1.07×10^{-8} to 3.53×10^{-8} m²/s, depending on the applied MP. Shrinkage percentage and bulk density of the samples varied from 1.2% to 4.9% and 3,440.77–1,091.41 kg/m³, respectively. Increase in the drying time and MP resulted in decreased antioxidant content occurring in first-order reactions. By the end of the drying period, TPC ranged from 14.69 to 10.66 mg GAE/g DW with an optimal MP of 300 W, while BCC and TFC ranged from 0.23 to 0.35 mg BCE/g DW and 20.71 to 23.51 mg CAE/g DW, respectively, increasing directly with MP. Higher values of ABTS scavenging capacity were obtained at higher MP. Overall, the MVD method has been proven to be an efficient technique for the drying of papaya seeds while retaining important phytochemical compounds.

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