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Kinetics of Mass Transfer, Colour, Total Polyphenol and Texture Change of Manilkara Zapota During Convective Air Drying

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The effects of air temperature and product size on drying kinetics, retained total polyphenol content (TPC) , hardness kinetics and total colour change (ΔE) kinetics of ciku (*Manilkara zapota*) were investigated. In addition, five terms of the theoretical model were used to estimate the effective diffusivity values during drying at temperatures from 40°C to 70°C. The drying rates of dried ciku were increased with increasing temperature and decreasing product size. It was found that hardness of ciku dried at temperature higher than 60°C increased significantly, when the moisture content was reduced to less than 0.5 g H_2O/g DM (dry basis). In terms of nutritional value, the retained total polyphenol content (TPC) of dried ciku in hot air drying also increasing with temperature. The highest retained TPC was 141 mg GAE/ 100g of samples, which can be obtained from drying at 70°C.

Keywords *Manilkara zapota*, Effective diffusivity, Hardness, Texture, Colour, Total polyphenol content.

INTRODUCTION

Ciku (*Manilkara zapota*) is a small ellipsoidal fruit native to Mexico and tropical America and now it is well spread throughout South East Asia and West India (Mickelbart 1996). It is enclosed with rough brown skin encloses a soft reddish-brown flesh with smooth black seeds and the diameter is ranging from 4.0 to 10.0 cm in diameter. Leong and Shui (2001) found that ciku has the highest antioxidant content among 27 types of fruit tested in their study. However, the TPC was reported to decrease with natural ripening (Mohamed et al. 1996, De Brito & Narain 2002, Shui et al. 2004). Therefore, the preservation of the fruit into dried product was applied in this study. Dying is a method of removing the water through evaporation and internal diffusivity. Much research has been carried out to investigate the isolation, structural elucidation, characterization, nutraceutical components and the changes of antioxidant of ciku during ripening stage (Shui et al. 2004, Ma et al. 2003, Kulkarni et al. 2007). However, no analysis was performed to investigate retained TPC of ciku after drying.

There has been some research on the drying characteristics of ciku samples. Ganjyal et al. (2003) found that vacuum-oven drying (14 to 31 hours) of ciku (*Achras zapota*, cv, Kalipatti), was better than convection air-oven drying (15 to 35 hours) at the temperatures tested (55^{O}C to 70^{O}C) in terms of drying duration; In sensory analysis, Jain and Jain, (1998) found that forced air (T<34^{O}C) dried ciku (*Achras zapota* L.) slices are preferred than sun-dried ciku (T<30^{O}C). Research on the retained TPC, as well as investigating the hardness kinetics, total colour change kinetics of ciku slabs are scarce.

The objective of this study was to investigate the drying kinetics and product quality of ciku. Determination of best fit model to predict the drying kinetics of ciku dried at different temperature (40^{O}C, 50^{O}C, 60^{O}C and 70^{O}C). Further, hardness kinetics, total colour changes kinetics and retained TPC at different temperatures were evaluated.

Theory

Semi-theoretical modelling

Semi-theoretical model curve fitting analysis performed in this study to predict the kinetics of ciku drying at different temperature. The models include the Newton or Lewis model, Page model, Henderson and Pabis model, Logarithmic model, Modified Page Equation II model, Simplified Fick's Diffusion model, Approximation of Diffusion model, Werma et al. model, Modified Henderson and Pabis model, Two Term Exponential model and Wang and Singh model (Liu & Bakker-Arkema 1997, O'Callaghan et al. 1971, Page 1949, Henderson & Pabis 1961, Yagcıoglu et al. 1999, Diamante & Munro 1991, Kassem 1998, Werma et al, 1985, Karathanos 1999; Sharaf-Elden et al. 1980, Wang & Singh 1978).

Effective diffusivities, effective diffusivity constant and activation energy

The effective diffusivity (D_{eff}), determined by using the analytical solutions of Fick's second law recommended by Sherwood and Newman (Coulson et al. 1987), is shown in Eq. (1). One-dimensional diffusion is applied in this study because of low thickness to width and length ratio (Thorvaldsson & Janestad, 1999). According to Zhou (2005), if two of the dimensions are much larger than the third dimension, the sample can be taken as a thin slab where only one dimensional mass transfer was significant along the thickness.

$$MR = \frac{8}{\pi^2}\{e^{-D_{eff}\,t(\pi/2l)^2} + \frac{1}{9}e^{-9D_{eff}\,t(\pi/2l)^2} + \frac{1}{25}e^{-25D_{eff}\,t(\pi/2l)^2} + \ldots\} \quad (1)$$

$$MR = \frac{M_t - M_e}{M_i - M_e} \quad (2)$$

Where M_i = initial moisture content, M_e= equilibrium moisture content and M_T = moisture content in actual time.

Five terms of the theoretical Eq. (1) were used to estimate the effective diffusivities (D_{eff}) during the falling rate period of drying. The effect of temperature on the effective diffusivity constant and activation energy can be described by the Arrhenius equation (3) (Madamba et al. 1996, Henderson and Pabis 1961, Crisp & Woods 1994).

$$D_{eff} = D_o \exp(-\frac{E_a}{RT_a}) \quad (3)$$

Where D_o= effective diffusivity constant (m^2/s), E_a = activation energy (kJ/mol), R = gas law constant (kJ/ kmol. K), T_a = air temperature (K)

EXPERIMENTAL METHODS

Samples preparation

Ciku was obtained from a local fruit supplier. The ciku peel and seed were removed and the flesh was cut into different dimensions by using a stainless steel knife, the dimensions were 2.0 cm x 3.0 cm x 0.5 cm, 2.0 cm x 2.0 cm x 0.5 cm and 2.0 cm x 1.0 cm x 0.5 cm.

Drying process

The samples were dried in an oven (Memmert, DO6836, Germany) at 40°C, 50°C, 60°C and 70°C. The velocity of the air approaching to the slabs was measured by an anemometer (Rotronic, D5-U-2, USA) and the average velocity was 0.965 ms^{-1}. The direction of the air flux was perpendicular to the oven door. Relative humidity and temperatures were measured using a hygrometer (HygroFlex, RS232, USA). The relative humidity were 24.4%, 12.8%, 7.2% and 4.6% corresponding to the drying temperatures tested at 40°C, 50°C, 60°C and 70°C.

The samples were removed at intervals during drying and weighed, before being returned to the oven. The weight loss of the samples was recorded using an Adventure OHAUS electronic balance (Model AR3130) with a range of 0-310($\pm$0.001g), at 15 minute interval for the first hour, followed by hourly intervals until equilibrium moisture content was observed.

Texture analysis

A ciku slabs were aligned horizontally on the platform and deformation on only one side of the ciku slabs was tested. The texture analyzer (TA.XT Plus, Stable Micro System, UK) fitted with a cylindrical puncture probe of 2.0 mm diameter was used. The texture analyzer was programmed so that the downward movement began at 10 mm above the surface of the sample. The probe was moved from the surface of the fruit to a depth of 30 mm (ciku slabs total thickness are 50mm) using a trigger force of 5.0 g with speeds of 1.0 mm/s, 5.0 mm/s and 5.0 mm/s at a pre-test, test and post speed, respectively. Hardness was automatically computed from each curve using the texture analyzer software macro (Texture Exponent 32 Stable Micro System, Texture Profile Analysis, 2007) (Adriana et al., 2006; Chong et al., 2008a; Chong et al., 2008b). The peak force was determined from the graph generated by the computer program.

Colour analysis

Colour analysis was performed using a colourmeter (AccuProbe, model HH06, USA). The changes in different colour attributes were calculated by Eq. (4), (5) and (6). The colour value L* expresses whiteness/ darkness in the ranges of 0 (black) to 100 (white). Whereas, b* and a* expresses yellowness (+60) /blueness (-60) and redness (+60) /greenness (-60), respectively. Higher positive value of (b*) correspond to more yellowness, while higher positive value of (a*) corresponds to more redness.

$$\Delta L^* = L^* - L_o^* \quad (4)$$

$$\Delta a^* = a^* - a_o^* \quad (5)$$

$$\Delta b^* = b^* - b_o^* \quad (6)$$

The total colour change (ΔE) is determined from Eq. (7) (Nsonzi & Ramaswamy 1998).

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{\frac{1}{2}} \quad (7)$$

Total polyphenol content (TPC) analysis

Dried ciku slabs were homogenized by using a blender (Mapporo, 800, Malaysia). 0.5g of homogenized ciku were weighted and extracted with 10ml acetone (70%). Then, the sample was further homogenized by placing it in the Ultrasonic bath (Sonicor, DSC-121TE, USA) for 30 minute. Homogenized sample was centrifuge for 15 minute at 5000 rpm using centrifuge (Hettich, Mikro 22R, Germany).

TPC of ciku extracts was determined using Folin-Ciocalteu reagent. Folin-Ciocalteu's phenol reagents, 2N, were purchased from Sigma-Aldrich (Selangor, Malaysia) Sdn. Bhd. Methanol, 98%, Gallic acid, 98%, Aceton, 99.5%, Sodium Carbonate, 99% and ethanol, 99.4% v/v were purchased from Fisher Scientific (Selangor, Malaysia) Sdn. Bhd. Firstly, 100ml of the extract was mixed with 0.5ml Folin-Ciocalteu reagent (previously diluted with 7.9ml of distilled water). Then, the mixture was mixed well and kept at room temperature (about 28°C) for 30 second. After 8 minutes, 300 μl of sodium bicarbonate was added to the mixture and mixed well. The mixture was left for 120 minutes at room temperature. The absorbance of the reaction mixture was measured at 765 nm using Spectrophotometer (Shimadzu, UV-2501PC, Japan). 100 ml of distilled water was used as a blank following the same procedure stated above. The TPC of the dried ciku was obtained by comparing with that of gallic acid via a calibration curve. Hence, the total polyphenol content in the extract was expressed as mg GAE (Gallic acid equilibrium) per 100 g sample.

The TPC of four commercial dried fruit was investigated using the same method. The purpose of this comparison is to find the best air-dried temperature and investigate the consumer acceptability ranged. Four varieties of dried fruits were purchased from local hypermarkets. The fruits comprised of mango (*Mangifera indica L.*), pineapple (*Ananas comosus*), kiwi (*Actinidia chinenesis*), and roselle (*Hibiscus sabdariffa Linn*).

Statistical analysis

These predicted values were calculated by non-linear regression analysis using Excel Solver program (Microsoft Office Excel, 2003). The goodness of fit between the predicted and experimental data was evaluated based on statistical analysis. High coefficient of determination (R^2) and low chi-square (χ^2), root mean square error (RSME) and mean bias error (MBE) indicating a good fit.

The results obtained from all quality analyses were analyzed in triplicate by using a completely randomized design. Analysis of variance (ANOVA) was performed by SAS statistical package (SAS Institute Inc, SAS/STAT 9.1, 2004). Means were compared by Tukey's Studentized Range (HSD) test at $p<0.05$.

RESULTS AND DISCUSSION

Effect of temperature

Figure 1 plots drying rate versus moisture content at different drying temperatures and product size. As the moisture content decreases, drying rate decreases. For ciku slabs with dimension of 2.0 cm x 3.0cm, the initial transient period occurred in the first 15 to 30 minutes. It was then followed by a falling rate period, where the drying rate is controlled by diffusion of moisture from the interior of ciku slabs to the surface.

The drying rates at the temperatures tested (70^{O}C, 60^{O}C, 50^{O}C and 40^{O}C) were in the range of 0.038 to 0.659 g H_2O/g DM. m^2. s, 0.371 to 0.596 g H_2O/g DM. m^2. s, 0.037 to 0.454 g H_2O/g DM. m^2. s and 0.0365 to 0.296 g H_2O/g DM. m^2. s, respectively. Highest drying rate of ciku slabs was 0.659 g H_2O/g DM. m^2. s found at a drying temperature of 70^{O}C after the initial transient period. This is because of a higher drying force (higher kinetics energy given by higher drying temperature). Furthermore, the drying rates of all the temperatures tested were relatively higher after the initial transient period because of the high moisture content and increase of product temperature. Similar observations have been reported in hot air drying of rosehip (Erenturk et al. 2004), chempedak (Chong et al. 2008a) and apple (Kaya et al. 2007).

The general drying characteristics and kinetics trend of ciku slabs 2.0 cm x 2.0 cm and 2.0 cm x 1.0 cm dried at different temperature as shown in Fig. 1 ranged from 40 to 70^{O}C were similar to product size 2.0 cm x 3.0 cm. In terms of drying rates, the drying rates of the ciku slabs increased with increasing drying air temperature. Referring to the three different product sizes tested, the smallest product sizes registered the highest drying rate (0.075 to 1.300 g H_2O/g DM. m^2. s) owing to bigger surface to volume ratio that eases the evaporation of the samples.

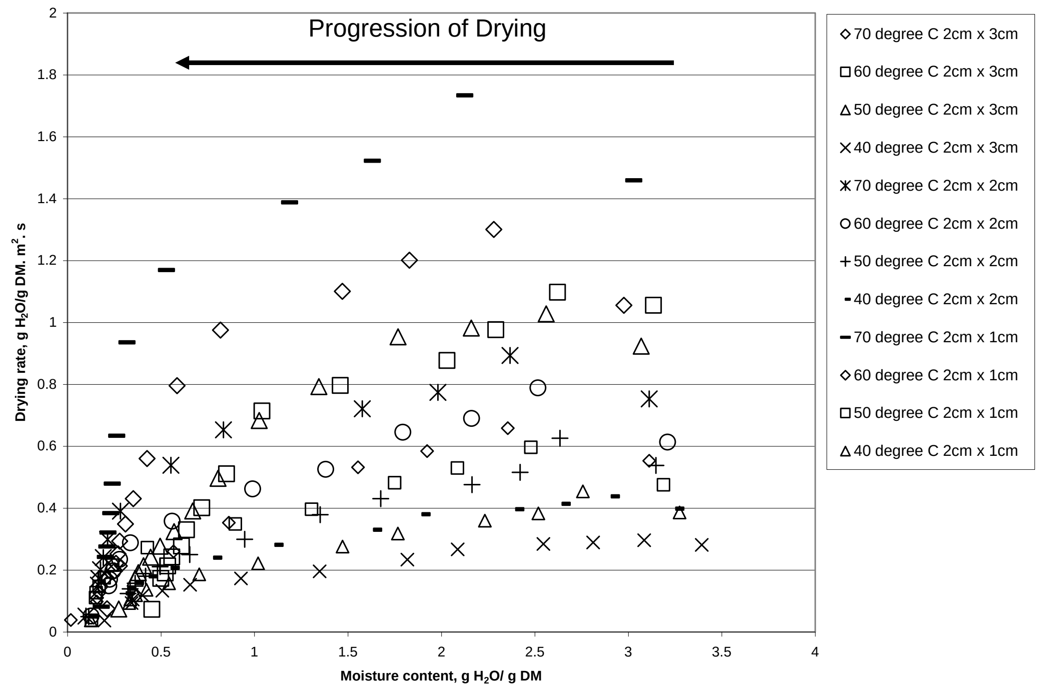

Figure 1. Effect of Temperature on Drying Rates During Hot Air Drying of Ciku with Slab Size of 2.0 cm x 3.0 cm x 0.5 cm, 2.0 cm x 2.0 cm x 0.5 cm and 2.0 cm x 1.0 cm x 0.5 cm

Curve fitting of semi-theoretical models

Eleven models were fitted to the experimental *MR* versus time profile of different temperatures. The statistical results showed that the Modified Henderson and Pabis model gave the highest coefficient of determination (R^2) and the lowest root mean square error (*RMSE*), mean bias error (*MBE*), chi-square (X^2) values.

Effective diffusivities of dried ciku slabs

Effective diffusivities of dried ciku samples at different air temperatures and product size are shown in Table 1. The effective diffusivities were in the range of 2.05 – 3.60 x 10^{-10}, 2.45 – 4.00 x 10^{-10}, 4.08 – 4.80 x 10^{-10} and 4.66 – 6.70 x 10^{-10} m^2/s at the temperatures tested (40^{O}C, 50^{O}C, 60^{O}C and 70^{O}C). The values fall within the range of diffusivity found in the drying of most agricultural products as shown in Table 2. Table 1 shows that the coefficient of determination (R^2) obtained from the predicted theoretical model and experimental data were in the range of 0.9635 to 0.9900.

Table 1. Effective Diffusivities of Hot Air Dried Ciku with Different Dimensions at Temperature Tested

Temperature, ^{o}C	Product Size	Effective diffusivities, m^2/s	R^2	RSME	χ^2
40	2.0 cm x 3.0 cm	2.05×10^{-10}	0.9732	0.0650	0.0045
50	2.0 cm x 3.0 cm	2.45×10^{-10}	0.9848	0.0464	0.0023
60	2.0 cm x 3.0 cm	4.08×10^{-10}	0.9837	0.0560	0.0034
70	2.0 cm x 3.0 cm	4.66×10^{-10}	0.9797	0.6008	0.0039
40	2.0 cm x 2.0 cm	2.15×10^{-10}	0.9932	0.0610	0.0040
50	2.0 cm x 2.0 cm	2.50×10^{-10}	0.9893	0.0471	0.0024
60	2.0 cm x 2.0 cm	3.75×10^{-10}	0.9900	0.0533	0.0031
70	2.0 cm x 2.0 cm	4.94×10^{-10}	0.9635	0.0634	0.0044
40	2.0 cm x 1.0 cm	3.60×10^{-10}	0.9820	0.0554	0.0033
50	2.0 cm x 1.0 cm	4.00×10^{-10}	0.9882	0.0547	0.0032
60	2.0 cm x 1.0 cm	4.80×10^{-10}	0.9743	0.0662	0.0048
70	2.0 cm x 1.0 cm	6.70×10^{-10}	0.9719	0.0656	0.0047

Table 2 Effective Diffusivity of Hot Air-Dried Fruits Reported in Literature

Fruits	Effective diffusivity (m^2/s)	References
Grape	$7.910 \times 10^{-10} – 2.500 \times 10^{-9}$	Doymaz & Pala 2002
Mulberry	$2.320 \times 10^{-10} – 2.760 \times 10^{-9}$	Maskan & Gogus 1998
Prune	$4.300 \times 10^{-10} – 7.600 \times 10^{-10}$	Sabarez, & Price 1999
Peach	$3.040 \times 10^{-6} – 4.410 \times 10^{-6}$	Kingsly et al 2007
Chempedak	$3.291 \times 10^{-10} – 4.534 \times 10^{-10}$	Chong et al 2008a
Red pepper	$5.010 \times 10^{-10} – 8.320 \times 10^{-10}$	Scala & Crapiste 2008
White radish	$6.920 \times 10^{-9} – 14.59 \times 10^{-9}$	Lee & Kim 2008
Blueberry	$2.240 \times 10^{-10} – 16.40 \times 10^{-10}$	Shi et al. 2008

Figure 2 shows the experimental and theoretical moisture ratio versus drying time for hot air dried ciku slabs. The logarithm of effective diffusivity (D_{eff})as a function of the reciprocal of absolute temperature (T_a) is plotted in Figure 3. A linear relationship between (ln D_{eff}) and ($\frac{1}{T}$) is obtained. The calculated diffusivity constants (D_o) were 3.62 x 10^{-7}, 4.14 x 10^{-6}, and 5.35 x 10^{-6} m^2/s for product with dimensions 2.0 cm x 1.0 cm, 2.0 cm x 2.0 cm and 2.0 cm x 3.0 cm, respectively. The activation energy (E_a) obtained were 18.14, 25.83, 26.56 kJ/mol, respectively based on the linearization of equation (3) as shown in Figure 3. The activation energy of the smaller samples was found lower than bigger samples. This is in agreement with the findings of Seyhan and Evranuz (2000) in drying of mushroom. The activation energy reported by Senadeera et al. (2003) for vegetable drying is in the range of 12.87 - 58.15 kJ/mol. Activation energy for hot air-dried red pepper, infrared-dried blueberries and vacuum-dried white radish were 23.35kJ/mol and 16.49 – 20.26 kJ/mol, respectively (Scala & Crapiste 2008, Shi et al., 2008, Lee & Kim, 2008).

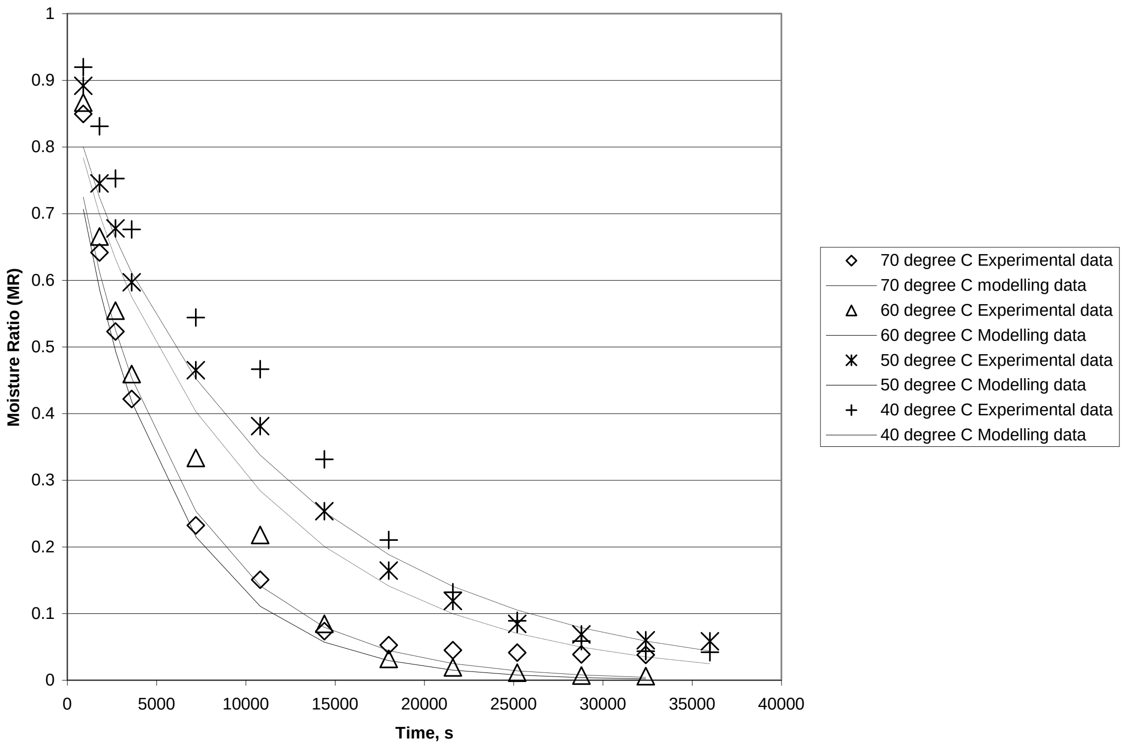

Figure 2. The Experimental and The Theoretical Modeling Data as A Function of Drying Temperature

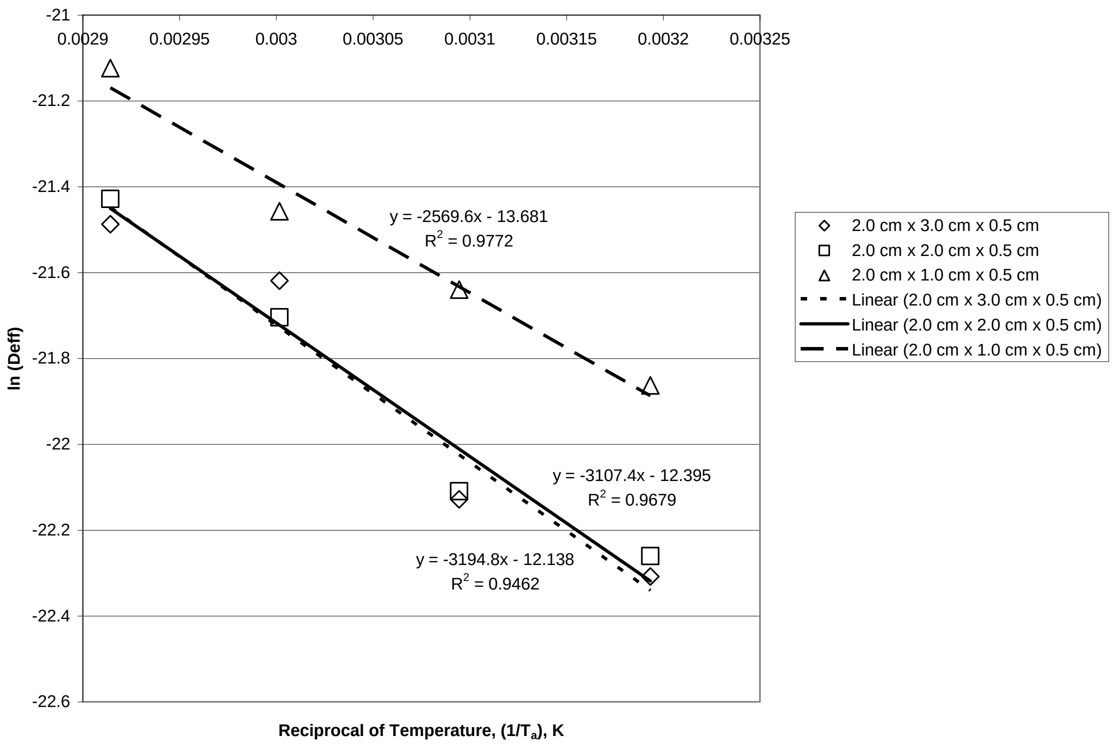

Figure 3. Correlation Between Effective Diffusivities and Temperatures

Kinetics of texture change

Figure 4 shows the hardness kinetics of ciku slabs dried at temperatures between 40°C to 70°C within 420 minutes. An increase in the hardness, which is a function of moisture content at different temperatures, can be observed in Figure 4.

Drying at 60°C and 70°C showed significant effects on hardness kinetics ($p<0.05$). This is because the hardness value increased appreciably when the moisture content was less than 20%, dry basis. The hardness of dried ciku samples changed from 94 g to 1105 g and 130 g to 1686 g, respectively. High temperature (higher driving force) drying of ciku samples promotes rapid removal of moisture from the samples and results in shrinkage and collapse of capillary voids inside the fruits. Similar finding was observed in drying of kiwi at high temperature, where fruit cell wall degraded (Femenia et al. 2008).

Ciku samples dried at 40°C and 50°C were found relatively soft compared to 60°C and 70°C. The hardness value increased from 71.0 g to 78.0 g and 84.6 g to 106.0 g, respectively. This is because of the slow moisture diffusion rate (low driving force), where the amount of moisture inside the samples is still very high at equilibrium moisture content compared to samples dried at 60°C and 70°C. Existing of internal moisture reduce the product hardness. This is similar to the findings of Medina-Torresa et al. (2008) in drying of nopal, who found that lower hardness value also obtained from lower drying temperature.

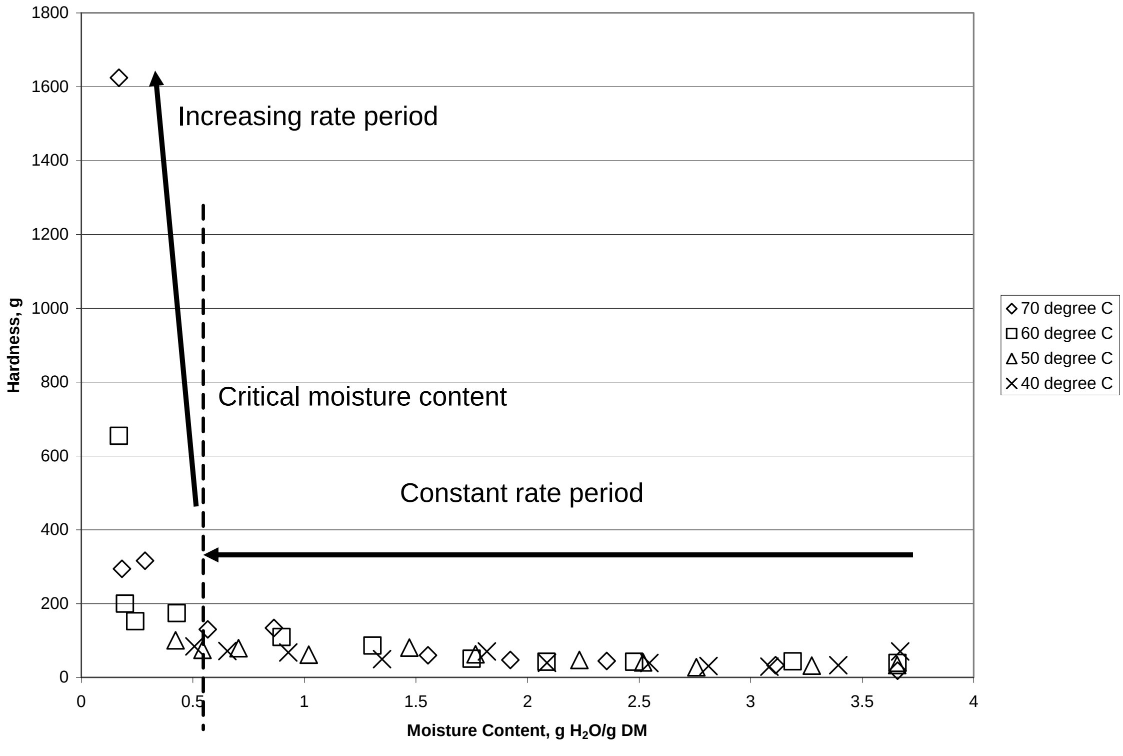

Figure 4. Effect of Temperature on Hardness During Hot Air Drying of Ciku Slabs

Kinetics of total colour change

Total colour change (ΔE) of ciku dried at various temperatures is given in Figure 5. The results indicate that the drying process led to darker products with less intense colour. The ΔE value increased with drying time (range between 15 minutes to 420 minutes) where it changed from 6.18 to 15.8, 2.22 to 15.2, 2.32 to 14.3, 1.25 to 13.7 for samples dried at 70°C, 60°C, 50°C and 40°C, respectively. Increment in

ΔE indicates colour change to a darker colour. A low positive ΔE value of ciku slabs were obtained at lower temperature. The results show that high dehydration temperatures in ciku drying also led to colour deterioration. This is because of non- enzymatic browning reactions and destruction of natural fruit pigments such as the decomposition of chlorophyll and carotenoid pigments. These findings are in agreement with the colour analysis of sun dried raisin (Kotsiopoulos & Saravacos 1995), thermal pasteurization of grapefruit juice (Lee & Coates 1999) and formation of brown pigment in hazelnut (Lopez et al. 1997) and microwave drying of banana (Maskan 2000). Higher drying temperatures result in bio-products undergoing greater colour change in drying of potato and carrot (Chua & Chou 2004). Total colour change values of dried fruits reported in literature were between 7 to 28 for dried banana and chempedak (Chua et al. 2001, Chong et al. 2008a).

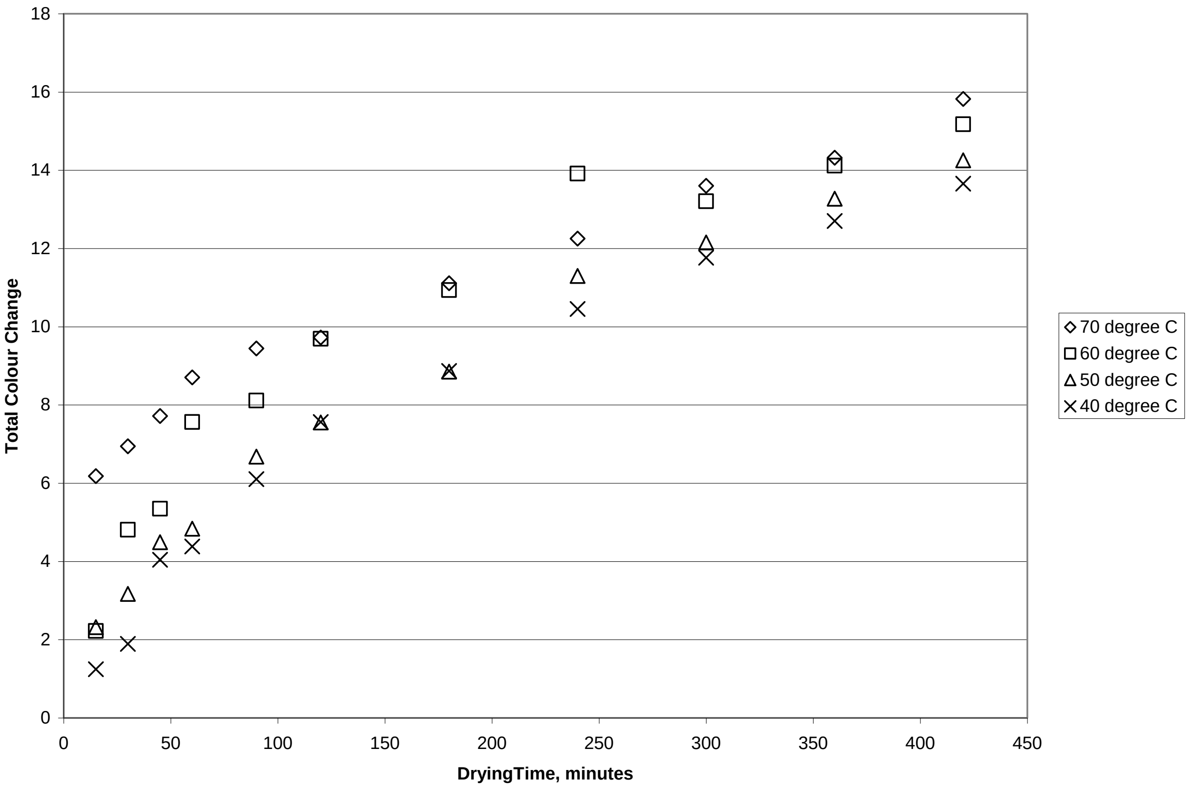

Figure 5. Total Colour Change of Ciku During Hot Air Drying at Different Temperatures

Total polyphenol content (TPC)

The quantity of total polyphenol content (TPC) retained in ciku after drying at different temperatures (40^{O}C, 50^{O}C, 60^{O}C and 70^{O}C) is shown in Table 3. The results revealed no significant difference ($p>0.05$) for all drying temperatures tested, even though TPC losses decreased with elevated temperature tested. A drying temperature of 70^{O}C recorded the highest TPC, which is 141 mg GAE/ 100 g of samples owing to the shorter drying time. This was in agreement with the findings of Yen & Huang (2000), where within a heating time up to three hours, the TPC of Hsian-Tao extract decreased about 50%. In addition, comparison between the dried ciku and commercial dried fruits shows that convective air dried ciku has the highest TPC. Four commercial dried fruits, mango, pineapple, kiwi, roselle TPC values were found in the range of 30 to 52 mg GAE/ 100 g of samples.

Table 3 Average Amounts of Total Polyphenol Content Retained in Dried Ciku (*Manilkara Zapota*)

Temperature, ^{O}C	Fresh Ciku, mg GAE/ 100 g of samples
40	90.0 ± 0.01 ab
50	90.0 ± 0.22 ab
60	92.0 ± 0.08 ab
70	141 ± 0.56 a
Mango	0.52 ± 0.08 b
Pineapple	0.3 ± 0.05 b
Kiwi	0.44 ± 0.27 b
Roselle	0.33 ± 0.09 b

The values indicate mean $\pm$ standard deviation from three replications. Values within the same column with similar letters are not significantly different.

CONCLUSIONS

Drying characteristics of ciku slabs exhibited an initial transient and falling rate period during hot air drying. The effective diffusivities calculated by using the theoretical model during falling rate period were in the range of 2.05×10^{-10} to 4.66×10^{-10} m^2/s. Based on the product quality analysis, future drying of ciku must be carried out at $70^{O}C$ because of higher retained TPC value. Texture of the convective air dried product can be control by performing finish drying at critical moisture content. The total colour change (ΔE) value was not significant as compared to other dried fruits values reported in literature. It was within the acceptable range of other dried fruits.

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NOMENCLATURE

DM	Dry matter
l	Thickness of the slab, m
t	Time, s
GAE	Gallic acid equivalent
L_o*	Initial $L*$ value
a_o*	Initial $a*$ value
b_o*	Initial $b*$ value

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