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Effect of temperature to the properties of sago starch

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Abstract. Recently, the importance of sago starch has increased, as it has become one of the main economically important agricultural crops to the most Southeast Asia countries. In the present work, an analysis on drying process of sago starch (*Metroxylon sagu* Rottb.) underwent various temperature has been made by using four empirical equations. The main goal of this analysis is to suggest the most accurate equation, in order to model and simulate the mechanical drying of sago starch. The experimental investigations were carried out in a gravity convection lab oven; and ± 50 g of sago starch (sample heights of 1 cm) was dried through four different temperatures, which were 50, 60, 70 and 80°C. The effect of drying temperature on the drying kinetics, as well as various qualities attributes of sago starch, such as microstructure, colour and functional properties were investigated. The results suggested that drying temperature has significant effect on sago starch drying kinetic; therefore, drying temperature would be the basis to select drying condition. Meanwhile, it was found that the various drying temperature ranging from 50 to 80°C affected the product quality especially in term of colour.

1. Introduction

Sago starch is the product of sago palm (*Metroxylon sagu*) or commonly called as “rumbia”. It is a tropical plant with huge trunk that may reach height up to 25 m and a diameter of 40cm; with pinnate-leaves up to 9 m long. It can tolerate wet-swampy lands such as peat soil. Sago palm reaches its maturity after 9 to 12 years of planting [5]. In term of productivity, sago palm produces the highest among the starchy crops in the world which is 250kg per palm; therefore it is also known as the ‘starch crop of the 21st century’ by many scientists [4]. Sago starch main content is carbohydrate which is higher than rice and wheat flour. For this reason, it has become the staple food since ancient times in



Asian region. In the food industry, sago starch has become an important raw material and it is predicted that the demand of sago in the future will increase. Malaysia produces among the highest starch in the world and Sarawak is the largest sago-growing areas in Malaysia [9].

Sago processing in modern factory starts by feeding a segment of sago log into a slicer to separate between pith and bark. Then, the debarked section is fed into a mechanical rasper to produce finer pieces, which are then fed in hammer mill via conveyor belt. The resulting starch slurry, which still containing coarse fibre, is passed through a series of centrifugal sieves. Further purification is done by the separation process in a nozzle separator through sieve bends, as well as a series of cyclone separators, to obtain very pure starch. Most importantly, dewatering of starch is carried out using a rotary vacuum drum dryer followed by a hot air drying process [5].

Meanwhile, traditional processing of sago is made manually from 'skinning' the bark to chopping the pith and grating it into material resembling sawdust. The pith is then kneaded with water and filtered through sieves to extract the relatively large (20–60 mm) starch granules. After being subjected to several washing and straining, the starch milk is allowed to run into troughs. The starch settles in the troughs, which is subsequently sun or kiln-dried.

Our previous study [13] revealed that the resulted starch from modern and traditional factory differ from each other in terms of moisture content, colour, microstructure, particle size and infrared absorbance. It is suspected that the differences are due to several factors such as the extraction and the drying process. Researches on correlation between extraction and starch yield have been done by some researchers[6, 11]. However, the drying mechanism of sago starch has not been studied, widely.

Drying is a crucial process in the food industry. In modern sago processing factory, the starch is dried to increase their shelf life, to reduce the packaging cost and to improve the appearance. However, some of the processing stages in the modern sago processing had changed the properties of the sago starch [7]. In the traditional factory, the quality of starch is far from demand but very close to its natural and beneficial properties. Therefore, this study aimed to understand the effect of temperature on the properties of sago starch.

2. Materials and Methods

2.1 Sample Preparation

Sago log were harvested from a nearby farm in Kota Samarahan, Sarawak. The log was kept in a refrigerator at a temperature under -80°C to avoid changes in its properties until the day it is ready for processing. The processing method involved chopping, blending, sieving and settling the starch.

First, the sago log was chopped with knife into small pieces. Then, the chunks were blended to produce sawdust. The sawdust was then mixed with water and sieved several times with kitchen siever, fabric siever (napkin) and lastly the 100um siever, respectively. After each sieving, the solution was left at room temperature for 3 hours, 1 hour and 30 min, respectively, before the precipitated starch can be collected and used in the drying experiment.

The sample preparation can be concluded as shown in figure 1.

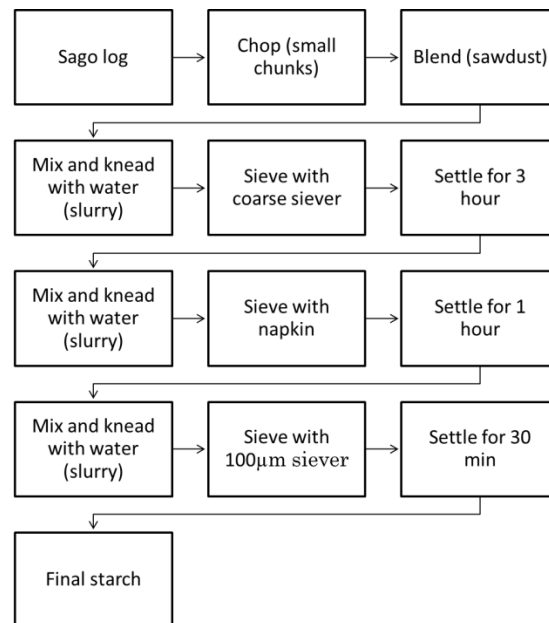


Figure 1. Step for extracting sago starch

2.2 Drying Experiment

A basic gravity convection oven was used for the drying experiment. For each experiment, the oven was preheated to the desired temperature. $\pm 50\text{g}$ of sago starch was placed in an aluminium foil-container and then it was placed in the oven. The treatment duration was dependent upon sample mass and oven temperature. The drying experiments were conducted at 50, 60, 70 and 80°C . The dried sago starch was weighed every hour until a constant mass was reached. During the sample mass measurement, the container was removed from the oven and it was placed on a weighing balance with least count of 0.001 mg, and then it was put back in the oven, quickly. This weighing process took place in less than one minute. Dried sample are ready for subjecting into psychochemical and Fourier transform infrared (FT-IR) analysis.

2.3 Mathematical Modelling and Data Analysis

Microsoft Excel 2010 version was used to analyse all drying data. All experiments were performed in three replications. The initial moisture content (M_i) was determined according to the AACC2000 method. The equilibrium moisture content (M_e) of the sago starch at various drying temperature was determined by following a dynamic method. The moisture content at which the sample weight was unchanged with drying time was the equilibrium moisture content. These values, together with moisture content at particular drying time (M) were used to calculate the moisture ratio (MR), as given in equation 1. This empirical model commonly used to evaluate the drying kinetics of food (T. Dikbasan, 2007).

$$\text{MR} = \frac{M - M_e}{M_i - M_e} \quad (1)$$

The moisture ratio (MR) was then subsequently regressed with Lewis model equation 2 to become equation 4.

$$\text{Lewis model, } \text{MR} = \exp(-kxt) \quad (2)$$

$$\text{Drying rate; } -\frac{dX}{dt} = k_d (X - X_e) \quad (3)$$

Here;

MR= (M-M_e) / (M_o-M_e); dimensionless moisture ratio

k_d= drying constant

t=drying time

The equation was integrated to give the following ones,

$$MR = \frac{X - X_e}{X_o - X_e} = \exp(-k_d t) \quad (4)$$

$$X = (X_o - X_e) \exp(-k_d t) + X_e \quad (5)$$

$$\text{Standard error, (SE)} = \left[\frac{\sum_{i=1}^n [(X_{Exp} - X_{Pred})^2]}{n-1} \right]^{\frac{1}{2}} \quad (6)$$

Where;

X_{Exp} = the Expth observed moisture ratio

X_{Pred} = the Predth moisture ratio

n = number of observations

These equations express first-order kinetics in terms of the moisture ratio equation (3) to give a predicted content or fitted moisture content, X, equation (4) that decreases exponentially with time. X_e is the equilibrium moisture content expressed on a dry basis. Standard error (SE) which means the deviation between predicted and experimental values was then calculated to confirm the model.

2.4 Physicochemical analysis

Physicochemical analysis involved the analysis of moisture content and colour analysis. The determination of moisture content in sago flour and lemantak were analysed by drying 3g of sample in gravity oven at 105°C until constant weight according to the AOAC, 2000 standard method. The moisture content was calculated in drying basis formula;

$$MC = \frac{M_i - M_t}{M_i} \times 100\% \quad (7)$$

Where M_i is the initial mass of sample (g) and M_t is the mass of sample (g) at a time (t), in this case, the final mass.

Meanwhile, an analysis of colour was carried out by taking the images of the samples manually, and analysed the images by using Matlab software that will give the pixel data of samples image in RGB value. The RGB values were then converted into CIE-L*a*b value by using a RGB-to-Lab converter program available from the internet at <http://colormine.org/convert/rgb-to-lab>. The parameter 'L' measures the luminosity of the sample from (L = 0) black to (L = 100) white. The parameters 'a' and 'b' are measured from -60 to +60. The degree of measurement for 'a' is from green to red, while 'b' is from blue to yellow. In another word, "L" represents lightness, "a" represents redness (+) or greenness (-), and "b" represents yellowness (+) or blueness (-). Whiteness index were calculated according to (Yadav et al., 2006).

$$WI = 100 - \sqrt{(100 - L)^2 + a^2 + b^2} \quad (8)$$

The initial colour of sago starch before drying was also measured and the value was used to calculate the total amount of colour change by using the following formula (Baini & Langrish, 2009).

$$E = \sqrt{(L_o - L)^2 + (a_o - a)^2 + (b_o - b)^2} \quad (9)$$

2.5 FT-IR

Fourier transform infrared (FT-IR) spectroscopy was used to evaluate the structural differences in the samples. The infrared spectra of the commercial and traditional sago starch were recorded on a Shimadzu Fourier Transform Infrared Spectroscopy (FTIR) 81001 Spectrophotometer. An appropriate amount of starch powder was pressed into a crystal window and the samples were analysed with a resolution of 4cm^{-1} and average scanning time of 1 min. Furthermore, the spectral resolution range of scan was 4000 to 400 cm^{-1} (mid-infrared region) with ATR mode being utilized. The obtained spectra were transferred into a data analysis package. All optical measurements were performed at room temperature under ambient conditions.

3. Results and Discussions

3.1 Equilibrium moisture content (EMC)

EMC is termed as the moisture content of a wet solid in equilibrium with air of given humidity and temperature. The EMC of sago starch drying in different temperature are shown in table 1. Drying sago starch at 50°C resulted in sago starch with highest EMC (10.36%), while drying at 80°C resulted in lowest EMC which is 1.31%. As for the whole result, the EMC was found to be decreased with the increase in drying temperature. Furthermore, the EMC value of starch drying at 50°C was significantly higher than that of 60, 70 and 80°C . This result can be explained due to decrease in the relative air humidity at higher temperature; in this case 60, 70 and 80°C [10].

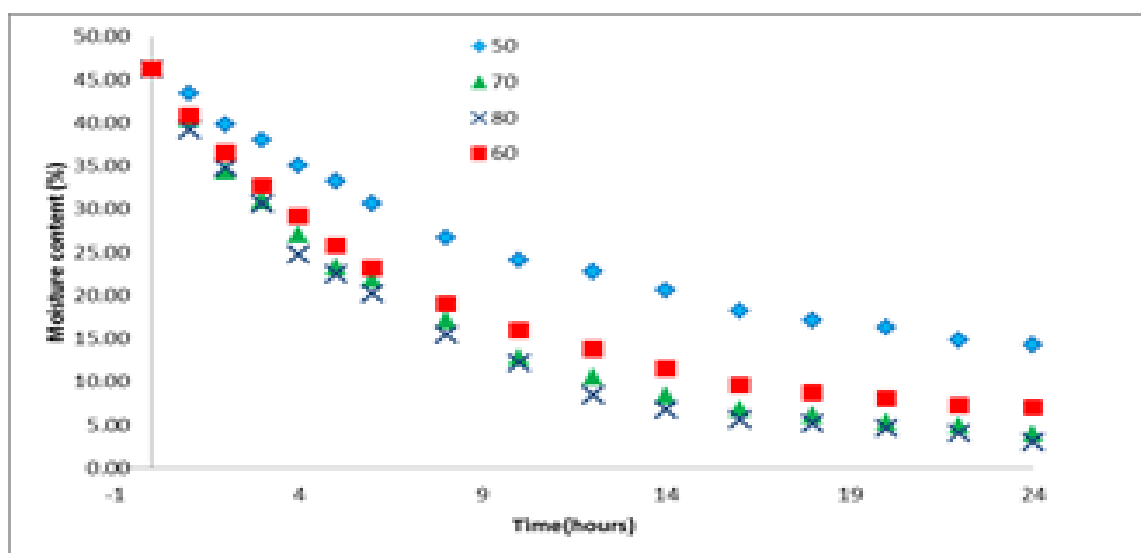
Table 1. Equilibrium moisture content (EMC) in dry basis (db) at different temperatures

Drying temperature, °C	EMC, % db ^a
50	10.36±0.10
60	3.45±0.14
70	2.47±0.13
80	1.31±0.18

^a All values are means of three measurements. Values are means ± standard deviation.

3.2 Drying Behaviour of sago starch

The changes in moisture content with time for different drying temperatures are shown in figure 2.

**Figure 2.** Moisture content of sago starch drying at different temperature (50, 60, 70 and 80°C)

The final moisture content of samples dried under different temperatures (50, 60, 70, 80°C) ranges from 14.30%, 6.99%, 4.01% and 3.07%, respectively. The result indicated that drying at higher temperature had produced sago starch with lower moisture content which suggested that drying temperature has an important effect on the final moisture content of sago starch.

In previous work, moisture content of commercial sago starch (rotary vacuum drum dryer and hot air drier at 50°C or above) had been compared with traditional sago starch (sun dry) which revealed that the former meet Malaysian standard (MS470 1992) for sago starch which is 13% maximum. On the other side, the latter did not meet the standard as the moisture content was 38.8% [7]. It should be noted that higher moisture content lead to high risk of microbial growth and spoilage while 0% drying until 0% moisture content will change the properties of the starch. In order to relate the previous result with this result, it is suggested that drying at temperature between 50 and 60°C will be the best to meet the standard for sago starch moisture content.

Figure 3 shows drying rate plotted over moisture content in different temperature (50, 60, 70 and 80°C). Sago starch display only falling rate period as many other foods and agricultural products which do not display constant rate period at all. This also indicates that the mechanism of mass transfer in sago starch is a moisture diffusion process. Therefore, during sago starch drying process,

the internal heat and mass transfer rates determine the rate at which water becomes available at the exposed evaporating surface. In addition, the drying surface becomes first partially saturated and then fully saturated until it reaches equilibrium moisture content [9].

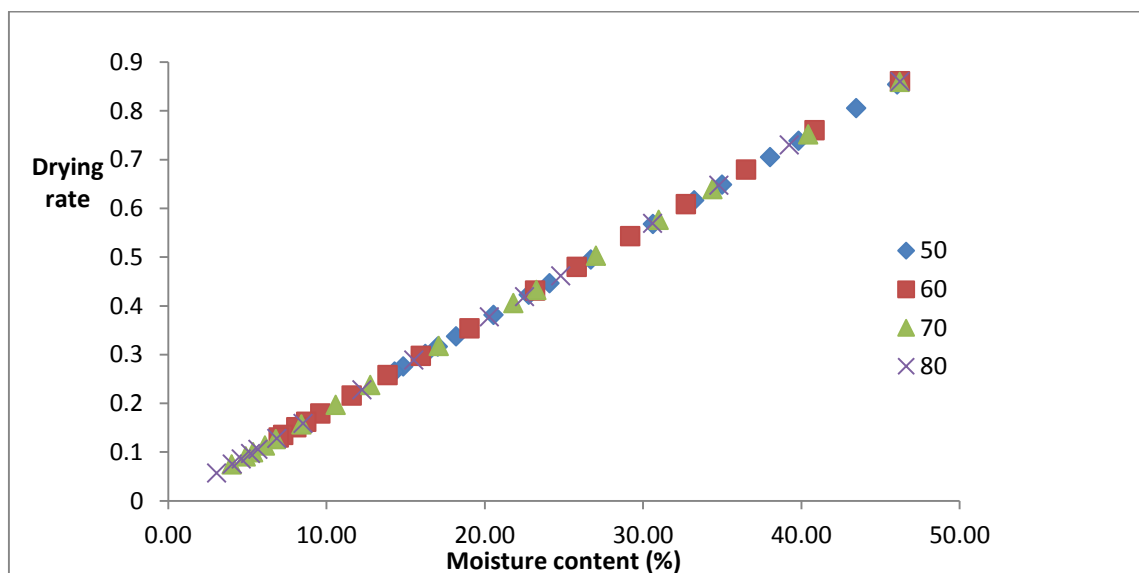


Figure 3. Sago starch drying rate over moisture content at different temperatures (50, 60, 70 and 80°C)

The drying rate of sago starch over time is shown in figure 4 below. It can be observed that at any of the time, drying curve at higher temperature is steeper than that at lower temperature, which means drying rate at higher temperature is higher than that of lower temperature, resulting in lower moisture content. The increase in drying temperature increases the heating rate and water vapour pressure inside the starch, thus accelerating the water migration inside it, consequently leading to higher drying rates and lower moisture content [3].

Besides, the result also shows that drying rate at 50°C decrease more gradually than at 60, 70 and 80°C are significantly steeper than 50°C. This shows that sago starch drying at 50°C exhibit consistent evaporation process for surface evaporation and inner moisture transportation.

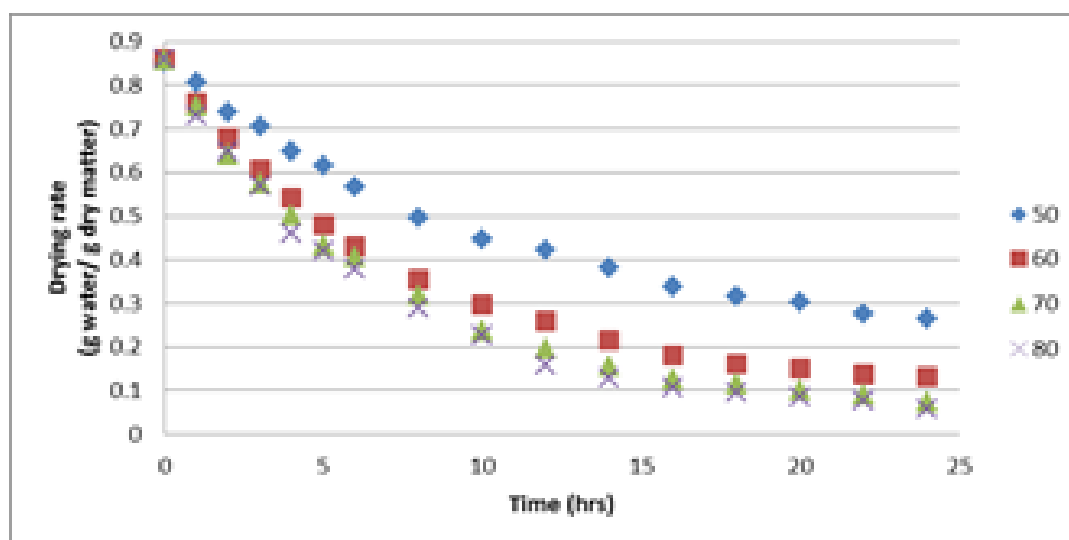


Figure 4. Sago starch drying rate at different temperatures (50, 60, 70 and 80°C)

Furthermore, the drying rate at 70 and 80°C are quite similar with each other indicated that sago starch reacts in the same manner when drying at both temperatures. Briefly, both temperatures allow high amount of near-surface moisture evaporation in the beginning of process. As the drying progresses, both drying rate decreases gradually because moisture is transported from the inner layer to the surface before it evaporates. As a result, the falling rate region expressed an increase in resistance to heat and mass transfer inside the material. In addition, rapid evaporation of surface moisture could lead to formation of hard layer surface which will inhibit the moisture removal from starch; thus, reducing the drying rate.

3.3 Mathematical Modelling

The first order approach, equation (2) by Lewis model was fitted to the experimental data of moisture ratio of sago starch at different temperatures. This model has been found to fit the experimental moisture content for materials that were dried continuously very well [12]. Standard error (SE) was used to determine the average difference that the experimental values fall from the regression line of Lewis model and the results are showed in table 2.

Table 2. Standard error (SE), correlation coefficient and drying constant results

Drying temperatures (°C)	Standard error (SE)	Correlation coefficient (R^2)	Drying constant (k)
50	0.77	0.9979	0.105
60	0.76	0.9993	0.144
70	0.63	0.9988	0.152
80	0.64	0.9981	0.162

The result from table 2 shows that low values of standard errors ($SE < 2.5$) were found for Lewis model. The average value also indicated that the experimental data were 0.7% closer to the regression line. In another word, 99.3% of the experimental data fall within plus/minus 2* standard error of the regression from the regression line, which is also mean quick approximation of a 99.3% prediction interval.

In order to confirm the relationship, a linear graph was generated between experimental and predicted data for each temperature as shown in Figure 3. The results showed that there were good agreements between the predicted moisture content from Lewis model and experimental values for all drying temperatures as shown in figure 5. All the R^2 values were considerably high ($R^2 > 0.98$) which indicated that this model was applicable for describing the drying behaviour of sago starch at drying temperature of 50, 60, 70 and 80°C.

In addition to this, the exponential curve between experimental and predicted data also shows good relationship in term of data fitting as shown in figure 6.

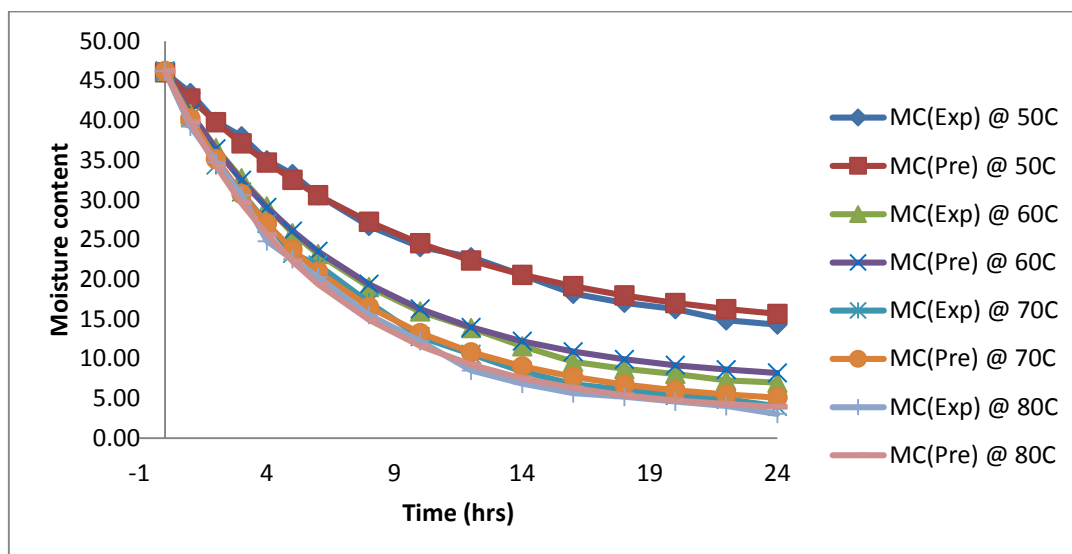


Figure 5. Comparison of experimental and predicted moisture content (mc) by Lewis model

The results obtained from figure 5 confirmed the applicability of Lewis model to be used for estimating drying behaviour of sago starch. In this preliminary work of drying sago starch, Lewis model as an empirical equation was selected to give better fit to the experimental data even though without any understanding of the transport process involved. Interestingly, this model has been used widely by many other researchers to predict the drying kinetic of fruits and vegetables [2].

3.4 Physicochemical Analysis

The effect of drying temperature on the properties of sago starch was observed through the colour changes analysis and the results are shown in table 3. As mentioned in Section 2.4, the parameter 'L' measures the luminosity of the sample from (L = 0) black to (L = 100) white. The parameters 'a' and 'b' are measured from -60 to +60. The degree of measurement for 'a' is from green to red, while 'b' is from blue to yellow. In another word, 'L' represents lightness, 'a' represents redness (+) or greenness (-), and 'b' represents yellowness (+) or blueness (-).

The results show that increase in temperature will increase the darkness (browning) of sago starch. Sago starches obtained from 50°C drying temperature showed more lightness (L) and whiteness (WI) as well as less browning than those dried at higher temperature. The changes in colour were relatively smaller, which suggested that drying sago starch at 50°C is suitable to preserve the natural colour properties of sago starch. In contrast, sago starches obtained from 80°C drying temperature showed more darkness (L) and browning than those dried at lower temperature. It is also can be observed that during the sago starch drying, the whiteness level decrease while the browning values increase in higher temperature.

Table 3. The measured final colours of sago starch dried at different temperatures

Temperature (°C)	Final colour parameters for sago starch drying ^a				
	WI	E	L	a	b
Predried	54.92	0	55.24	-0.18	5.38
50	52.02	3.95	52.77	-0.27	8.47
60	48.24	8.22	49.47	-0.17	11.23
70	44.13	12.24	44.59	5.76	4.22
80	38.78	16.80	39.82	4.11	10.48

^a WI=whiteness; E=browning; L=lightness; a=redness; b=yellowness

In previous study [7], the whiteness value of commercial sago flour was significantly higher than traditional sago flour even though it has been processed with high temperature (rotary vacuum drum dryer and hot air drying). This can be explained with the bleaching treatment applied in commercial sago flour factory [8]. However, in this study, it is confirmed that drying sago starch in high temperature result in unwanted color changes or the degradation of starch color. In fact, the higher the temperature, the darker the color.

Browning of sago starch is linked to its enzyme activity of isolated-DL-epicatechin and D-catechin compounds. These polyphenol compounds are hard to remove during extraction. The consequent activity of polyphenol oxidase will lead to color changes. The amount of (+)-catechin and (-)-epicatechin oxidized will increase when mix with water containing iron and copper, especially unfiltered water. Furthermore, the amount of these polyphenol compounds will also increase with an increase in temperature above 30°C [5].

3.5 Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR spectra of sago starch in different drying temperature, including pre-dried, are shown in figure 7. The x-axis represents wavelength with range of 4000-600 cm⁻¹ region and the y-axis represent percentage transmittance which show absorbance value or intensity.

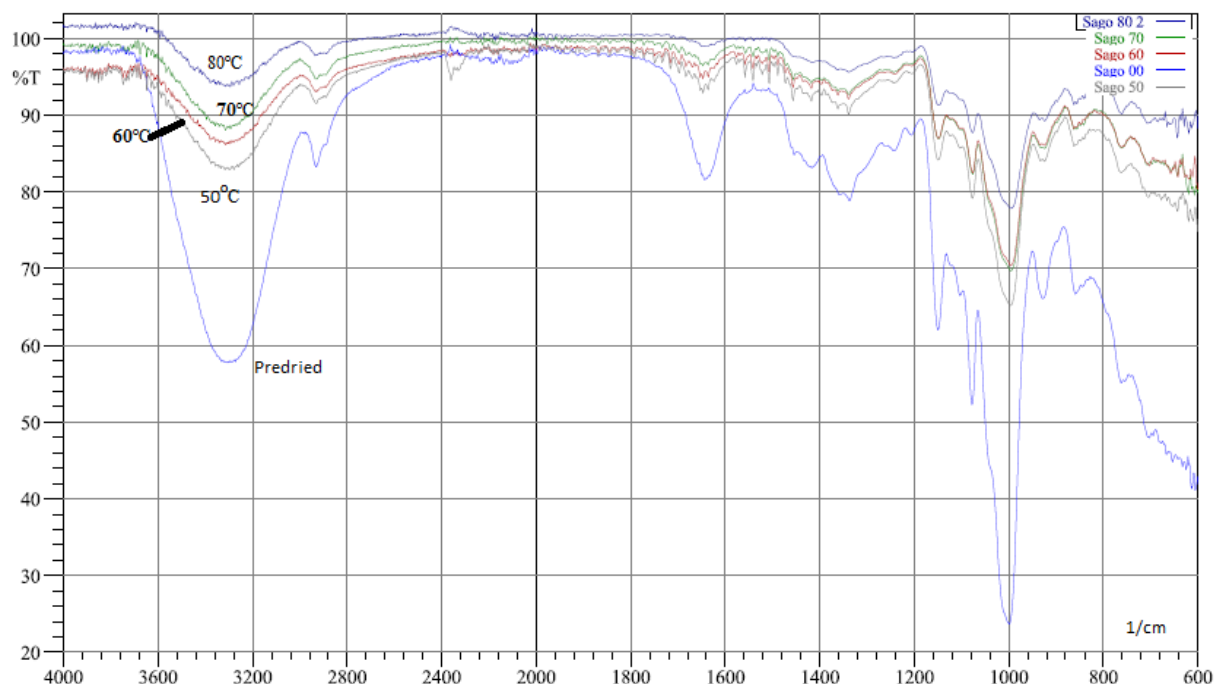


Figure 6. FT-IR spectra of sago starch drying at different temperature 50, 60, 70 and 80°C

The result shows that the pre-dried, as well as all dried starches exhibited a similar pattern and showed 11 major peaks. The absorbance peaks are near 760, 880, 960, 1000, 1080, 1160, 1360, 1440, 1640, 2880 and 3240 wavelength. Peak absorbance in starch have been assigned and matched with the vibrational modes of the chemical bonds and the structures of starch molecules.

Table 4. Peaks absorbance and the assigned chemical bonds/structures [14]

Peak modes (result) cm^{-1}	Peak attribution
3240, 2880	O-H and C-H bond
1640	H ₂ O bending vibration
1440	H-C-H, C-H and O-H bending modes
1160, 1360	C-O-H stretching
1080	C-O bond stretching
1000	The ordered and amorphous structures of starch
960	D-glucopyranosyl ring vibrational modes
880	C-H absorbances of the D-glucopyranosyl rings
760	D-glucopyranosyl ring stretching

Peaks absorbance in figure 6 and their attribution are listed in table 4. These 11 peaks absorbance play crucial role for the main structure of starch which is O-H and C-H bond, as well as the ordered and amorphous structures of starch. Therefore, the change in size of FTIR peaks may indicated either the change amount of bonds exist in the sample or the difference in polarity. Besides, it may also indicated the presence of asymmetric stretching bond that has the most energy, yet unstable.

Result from figure 5 shows that there were significant differences of peak intensities and width between pre-dried and dried sago starch which also can be observed at all the major peaks. Dried sago starch exhibited significantly less absorbance width and intensity compared to pre-dried starch, possibly due to oxidation during drying process. It also can be observed that the peaks width and intensities decrease with increase in the drying temperature. Increase in temperature may increase the oxidation, thus explained the decreasing in peak width and intensities.

4. Conclusions

In this study, the drying kinetics and its effects of drying temperatures (50, 60, 70 and 80°C) on sago starch were investigated. The results obtained showed that drying at higher temperature produced sago starch with lower moisture content. It is suggested that drying temperatures has an essential role in the characterization of sago starch drying behaviour in terms of equilibrium moisture content and drying kinetics. Lewis model was found to fit the drying of sago starches very well. Drying process also had changed the colour and chemical bonding in sago starch. Increase in temperature resulted in darkness increment. However, since drying process is one of a crucial process in sago starch industry, it is suggested that drying at 50°C will be the best temperature in terms of starch quality of sago starch.

Acknowledgements

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