



Faculty of Engineering

**Study on the Properties and Drying Kinetic Analysis of Sarawak Sago
Starch**

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Study on the Properties and Drying Kinetic Analysis of Sarawak Sago Starch

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DECLARATION

I declare that the work in the thesis was carried out in accordance with the regulations of Universiti Malaysia Sarawak. Except where due acknowledgments have been made, the work is that of the author alone. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any degree.



.....

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ABSTRACT

The study covers the properties, drying kinetics, and drying models of Sarawak sago starch. Properties analyzed include the composition, morphology, size, color, viscosity, chemical bond and thermal stability. The properties were compared for sago starch produced from different sources. The first sample was the commercial sago flour, bought from Kota Samarahan supermarket, the second sample was lemantak, bought from local producer, which was prepared using traditional method, and the third sample was prepared in the laboratory. Effect of drying was analyzed using the sago starch that was prepared in the laboratory, dried using an oven at temperatures of 50 to 80°C and fan speed of 0 to 0.95 m/s, microwave at 200W, freeze dryer at -92°C, and sun drying. In addition, the effect of sample thickness of 0.5 to 2 cm, and different location within the oven was also assessed. For oven drying, sago starch was dried continuously and intermittently; continuous drying refers to drying without interruption, and for the intermittent drying, a drying schedule that involves switching the oven 'on' and 'off' for certain period. The drying models assessed were thin layer models of empirical concept, simplified Fickian model and Characteristic Drying Curve concept. Results show that there were differences in the measured properties for sago starch prepared using different methods. It was found that lemantak and starch prepared in the laboratory have quite similar properties, but quite significant differences observed for the commercial sago flour, which has lower in total starch content of 29.79%, resistant starch content of 8.19% , viscosity of 7.45% as well as broken granules. Drying at 60°C for 15 hours increased the whiteness and thermal stability and decreased the moisture content of the sago starch. Increase in whiteness was observed based on increment in whiteness index of 46.89%, while moisture content decrease of 75%. It was also found that the final

moisture content of samples dried intermittently was higher than those dried continuously, but below than 13%, which is considered within the quality required. The intermittent oven drying is considered more effective drying than continuous drying in terms of energy saving; the drying period for intermittent drying (10.8% at 12 hour) was almost similar with the period of continuous drying (9.2% at 12 hour). The use of fan was found to increase the drying process. It was observed that drying at lower temperature of 60°C with fan velocity of 1 m/s gives estimated diffusivity of $5.77 \times 10^{-9} \text{ m}^2/\text{s}$, which is higher than that observed for drying at 80°C without fan gives estimated diffusivity of $1.27 \times 10^{-9} \text{ m}^2/\text{s}$, suggesting that drying at lower temperature with added fan can be a good option for drying starch. It was also found that lower sample thickness results in faster drying. The diffusivity for sample thickness of 0.5 cm was estimated to be about $8.2 \times 10^{-10} \text{ m}^2/\text{s}$, which is 200% higher than that estimated for sample thickness of 2 cm with diffusivity of $2.5 \times 10^{-9} \text{ m}^2/\text{s}$. Meanwhile, the sample position within the oven showed insignificant drying effect. Most drying models able to illustrate the drying kinetics of starch satisfactorily except drying characteristics curve model. The empirical model gives the lowest standard error of 0.006. The fitted parameters for drying at 50 to 80 °C were drying constant estimated between 0.043 to 0.225, diffusivity between from 7.086×10^{-10} to $1.268 \times 10^{-9} \text{ m}^2/\text{s}$ and activation energy of 17.7 to 19.4 kJ/mol.

Keywords: Sago starch, starch properties, drying kinetic, drying profile and model fitting

Kajian terhadap Ciri-ciri Semulajadi dan Kinetik Pengeringan Kanji Sagu Sarawak

ABSTRAK

Kajian ini meliputi sifat, kinetik pengeringan, dan model pengeringan pati sagu Sarawak. Sifat yang dianalisis termasuk komposisi, morfologi, saiz, warna, kelikatan, ikatan kimia dan kestabilan terma. Analisis sifat pati sagu yang dihasilkan daripada sumber yang berbeza juga termasuk proses tradisional pati sagu (lemantak), tepung sagu komersial, dan pati sagu basah yang diproses secara makmal. Eksperimen pengeringan lanjut melibatkan kanji sagu basah yang diproses di makmal yang telah dikeringkan menggunakan kaedah berbeza termasuk pengeringan ketuhar berterusan, pengeringan ketuhar terus-sekejap, pengeringan matahari, pengeringan gelombang mikro dan pengeringan beku. Untuk pengeringan ketuhar, suhu yang digunakan adalah antara 50 hingga 80°C, kelajuan kipas 0 hingga 0.95 m/s, ketuhar gelombang mikro pada gelombang 200W, pengeringan beku dan pengeringan matahari. Sebagai tambahan, ketebalan sampel 0.5 hingga 2 cm, dan lokasi yang berbeza di dalam ketuhar juga dianalisa menggunakan eksperimen pengeringan ketuhar. Untuk pengeringan berterusan, kanji dikeringkan secara berterusan tanpa gangguan. Manakala untuk pengeringan sekejap-sekejap, jadual pengeringan melibatkan pensuisan 'hidup' dan 'mati' ketuhar. Model pengeringan yang dinilai adalah model lapisan nipis konsep empirikal, model Fickian dipermudahkan dan konsep Lengkung Pengeringan Ciri. Keputusan menunjukkan bahawa kaedah pemprosesan kanji sagu mempengaruhi sifat kanji sagu. Didapati kanji komersial mempunyai jumlah kandungan kanji yang lebih rendah iaitu 29.79%, kandungan kanji tahan 8.19%, kelikatan 7.45% serta butiran pecah berbanding kanji yang disediakan di makmal. Selain itu, pengeringan pada suhu 60°C selama 15 jam menunjukkan peningkatan keputihan dan kestabilan terma, serta penurunan

kandungan lembapan. Peningkatan keputihan diperhatikan berdasarkan kenaikan indeks keputihan sebanyak 46.89%, manakala kandungan lembapan menurun sebanyak 75%. Ia juga didapati bahawa kandungan lembapan akhir sampel yang dikeringkan secara berselang-seli adalah lebih tinggi daripada yang dikeringkan secara berterusan, tetapi di bawah 13%, yang dianggap dalam kualiti yang diperlukan. Pengeringan ketuhar sekejap dianggap pengeringan yang lebih berkesan daripada pengeringan berterusan dari segi penjimatan tenaga; tempoh pengeringan untuk pengeringan sekejap (10.8% pada 12 jam) adalah hampir sama dengan tempoh pengeringan berterusan (9.2% pada 12 jam). Juga didapati bahawa keresapan pengeringan pada 60°C dengan halaju kipas 1 m/s (5.77×10^{-9} m²/s) adalah lebih tinggi berbanding dengan pengeringan pada 80°C (1.27×10^{-9} m²/s), mencadangkan pengeringan pada suhu yang lebih rendah dengan kipas tambahan boleh menjadi pilihan yang baik untuk mengeringkan kanji. Ketebalan sampel yang lebih rendah menghasilkan pengeringan yang lebih cepat; pemerhatian menunjukkan peningkatan difusi sebanyak 200% untuk ketebalan sampel daripada 2 cm (8.2×10^{-10} m²/s) kepada 0.5 cm (2.5×10^{-9} m²/s). Sementara itu, kedudukan sampel dalam ketuhar menunjukkan kesan pengeringan yang tidak ketara. Kebanyakan model pengeringan dapat menggambarkan kinetik pengeringan kanji dengan memuaskan dengan model empirikal memberikan ralat piawai terendah iaitu 0.006, kecuali model lengkung ciri pengeringan. Parameter yang dipasang untuk pengeringan pada 50 hingga 80 °C ialah pemalar pengeringan yang diperolehi antara 0.043 hingga 0.225, keresapan antara 7.086×10^{-10} hingga 1.268×10^{-9} m²/s dan pengaktifan 17.7 hingga 19.4 kJ/mol.

Kata kunci: Kanji sagu, sifat kanji, kinetik pengeringan, profil pengeringan dan pemasangan model

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LIST OF ABBREVIATIONS

ASEAN	The Association of Southeast Asian Nations
AACC	American Association of Cereal Chemists
ACH	Air Changes per Hour
ANOVA	One-way Analysis of Variance
AOAC	Association of Official Analytical Chemists
ATR	Attenuated Total Reflectance
BU	Brabender Unit
CIE	Commission on Illumination
EMC	Equilibrium Moisture Content
FTIR	Fourier Transform Infrared Spectroscopy
NaHSO ₃	Sodium Hydrogen Sulphite
RGB	Red Green Blue
RS	Resistant Starch
SD	Standard Deviation
SE	Standard Error
SEM	Scanning Electron Microscope
SMEs	Small Medium Enterprises

SO ₂	Sulphur dioxide
TGA	Thermogravimetric
UV	Ultraviolet
XRD	X-ray Diffraction

LIST OF SYMBOLS AND UNITS

$^{\circ}\text{C}$	Degree celcius
m/s	Meter per second
cm	Centimeter
m	Meter
g	Gram
L	Luminous
a	Redness
b	Yellowness
ppm	Parts per million
μm	Micrometer
MR	Moisture Ratio
M_t	Moisture content at a time
M_i, X_o	Initial moisture content
M_e, X_e	Moisture content at equilibrium
k_d	Drying constant
t	Time
X, MC	Moisture content

a, b, c	Drying parameter
d_p	Diameter of dried material
a_w	Water activity
u	Air velocity
kJ/mol	kilojoule per mole
DR	Drying rate
N_v	Unhindered drying rate
ϕ	Characteristic moisture content
X_{cr}	Critical moisture content
D_r	Pre-exponential factor
D_{EF}	Activation energy
T	Temperature
n	Number of data
ml	Mililiter
nm	Nanometer
x	Number of times
WI	Whiteness index
BI	Browning index

w_i	Initial weight
w_t	Weight at a time
w_e	Weight at equilibrium
p	Probability value
R^2	Correlation coefficient
cm^{-1}	Wavenumber
$w.b.$	Wet basis
$d.b.$	Dry basis
$T\%$	Transmittance
e	Exponential
h	Thickness

CHAPTER 1

INTRODUCTION

1.1 Background

1.1.1 The Importance of Sago Starch Industry in Malaysia

Sago starch has become a staple food for people around Southeast Asia since ancient times. Similarly, Penan and Melanau people in Sarawak especially have been using lemantak in their daily life since long time ago. Their main dish is made from lemantak and is called Linut. It is prepared by mixing hot water and stirred to form a clear jelly-like dough. It can be consumed together with hot and spicy protein-based side dishes called “sambal”. Lemantak also can be used to make sago pancake or commonly known as “tumpik”. Tumpik is a kind of baked starch cake, mixed with shredded coconut, and topped with protein-based food such as fish and prawn. Besides, lemantak also can be used to make others traditional food such as cucur lemantak, pais lemantak, crackers and some types of Melanau sweet traditional delicacy. The demand of lemantak by local people creates high potential of market in local food industries, gives opportunity for domestic market to grow, and of course indirectly can give better economic impact to the small business operators.

Today, it has become an important raw materials in food industry, especially in Asian region. In the food industry level, it is predicted that demand of sago in the future will increase as it has become an important raw food material. Sago starch is not only used as food, but also used in the processing of glue and cosmetics (Singhal et al., 2008). Hence, understanding the characteristics of sago starch can be important in determining its

appropriate applications. For example, the pasting profile of sago starch was studied in order to determine its usage in producing qualified noodle (Purwani & Thahir, 2006).

Malaysia is one of the world's leading sago producer, while Sarawak is the largest sago-growing areas in Malaysia. The list of annual sago starch production from world's top sago producer is given in Table 1.1 below.

Table 1.1: Annual sago production from Indonesia, Malaysia, and Papua New Guinea as the world's top sago producer (Trisia et al., 2021)

Country	Sago starch produced
Indonesia	75000 – 87000 tons
Malaysia	41000 – 51000 tons
Papua New Guinea	28200 – 66500 tons

Moreover, Sarawak had exported about 37,884 tons of sago starch worth RM78.3 million in 2021 as reported by Jack (2022). According to the data obtained from The Official Portal of Sarawak Data, Sarawak has exports sago starch to many places and countries such as to the Peninsular Malaysia, Japan, Singapore, Thailand, Vietnam, Myanmar, China, and Indonesia. Table 1.2 below shows income generated through Sarawak sago starch exports, which shows significant impact or contribution of sago processing to Sarawak's economy.

Table 1.2: Income generated by Sarawak from sago exports

Year	Quantity (Tons)	Value (RM)
2008	44,424.75	57,067,424
2009	41,484.64	60,403,459
2010	44,448.83	62,831,429
2011	51,014.19	91,432,141
2012	48,152.52	81,106,560
2013	48,289.55	77,018,639
2014	46,955.67	81,008,758

Before 1908s, there were about 40 sago traditional mills operated in Sarawak to export sago starch. By the late 1980s, ten modern sago factories have been established and they are still operating until today. These modern factories had caused the traditional sago mills to closed down due to market competition (Jong, 2018). The small-scale traditional sago processing plants operating today only produce sago starch for the local market in Sarawak.

Sarawak is the only state in Malaysia known to produce sago starch both commercially with modern techniques and traditionally for export and local needs, respectively (Shin & Collins, 2015). Sago starch prepared with modern techniques commonly referred to as sago flour, while sago starch prepared traditionally referred to as 'lemantak'. The differences in the processing techniques for both types of sago starch have

influenced the characteristics of sago starch produced. Lemantak has been categorized as second grade sago flour due to its low quality compared to the first-grade commercial sago flour. This condition had affected the sales and the income traditional sago producers (Siti Mazlina et al., 2007). Though, the production of lemantak is socioeconomically important for Sarawak's economic growth. Various food businesses related to lemantak have been recognized by the government of Sarawak as one of the Small and Medium Enterprises (SMEs) industries. Lemantak is suitable for SMEs as sago palm can produce high amount of starches with low cost production process (Mad Nasir et al., 2011).

1.1.2 The Differences between Lemantak and Sago Flour

Sarawak is the only state in Malaysia known to produce sago starch both commercially and traditionally, by using modern techniques and traditional method, respectively (Shin & Collins, 2015). The commercial sago starch is produced in modern factories, here, referred to as sago flour, while the traditional sago starch is produced by local people, here, referred to as lemantak. In Sarawak, there are three large sago plantation areas that are Dalat Sago Plantation, Mukah Sago plantation and Sebakong Sago Plantation. There are also 21 sago processing plants to produce commercial sago flour for domestic and export (Michiel, 1997).

In general, sago starch extraction involved chopping and rasping the sago log, sieving, and settling the starch slurry and finally drying the starch sediment. The principles and methods in commercial sago processing are similar to the methods used in traditional processes but differ in terms of equipment's used, quantity of raw material, surrounding condition and human resource. These differences are considered as some of the factors contributing to the differences between the quality of modern and traditional sago starch. In

relation to that, the quality of sago starch depends on several factors such as the type of raw material, quality of water used during the process, extraction method, drying process, packaging, and duration of transportation. These qualities will determine the achievable market value, the possible end user, and its suitability to be used in downstream biotechnological industries.

Sago processing in modern factories begins with 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 contains coarse fiber, is made to pass through a series of centrifugal sieves. Further purification is still needed by separation 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 hot air drying (Karim et al., 2008).

Meanwhile, sago mill focused on the extraction of sago starch as a small business in Sarawak to produce lemantak for domestic market. In this traditional method, the ‘skinning’ of the pith is done manually by axe. The un-skinned pith is then chopped and grated into sago-sawdust. The sago-sawdust is then kneaded with water and filtered through sieves to extract the starch while discarding the dust. White slurries are obtained after this process. This white slurry is then subjected to several washing and straining. The starch milk is allowed to run into troughs while flowing out the excess water. The starch will settle in the troughs. Finally, the settled starch is dried under the sun for a few days until the starch is good enough for packaging (Singhal et al., 2008). In the traditional method, the pith is grated while in the modern factories, mechanical rasper is used to get finer sago particles, which may affect the final sago starch produced.

According to Kopli (2015), sago flour produced from modern factories in Sarawak was dried through quick hot drying of starch slurry. In addition, some additives were added to the starch such as preservatives and color bleach. The hot quick drying may require higher energy use, but shorter time, while the additives and bleach results in starch with evenly white color that has long shelf life. However, lemantak that are prepared traditionally is still gaining high demand among local people in Sarawak since it has the original and fresh taste as compared with sago flour. The unique taste and properties are important for meals such as linut and tumpik as their simple preparation method causes a sensitivity of taste to the lemantak, in which fresh and original taste is deserved.

1.1.3 Drying Process

Drying process is one of the most crucial parts in the food industry and most of the product-based industries such as paper, wood, and textile. Drying means the process of reducing moisture content in materials through heat and mass transfer processes. In foods industries, the end products are subjected to heat that removes its moisture content down to the lowest point that will stop microbe activities. Moisture in foods encourages the spread of microbe activities that spoil the foods. In addition, dried foods are much more easily packed, reduces their transport weight, prevents spoilage, and consequently reduce food wastage. According to Sagar (2010), more than 20% of the world's perishable crops are dried to increase their shelf-life and promote food security. The challenge in food drying is to get an appropriate drying technique that uses minimum heat usage as well as maintaining the quality of the food; different materials may have different drying behavior and characteristics.

There are several methods for food drying process such as solar (open-air) drying, sun drying, smoking, convection drying, drum drying, hot air drying, spray drying, fluidized bed drying and freeze drying. Different drying conditions affect the properties and quality of dried material in different manners. Therefore, one important area in drying process is to find the best approach of drying to get the desired quality of dried material. Sun drying, a common cheap drying method, is always used by small industry to produce local dried food, but it has disadvantages of producing products with inconsistent quality due to non-uniform drying. This is because drying happens fast during sunny hours and slow drying is expected during cloudy or rainy days. Moreover, sun drying is time-consuming, labor demanded and highly exposed to environmental contamination such as pests, rats, and microorganisms.

Solar (open-air) drying is an alternative to sun drying which is commonly done within an enclosed solar dryer that also possibly improve the disadvantages such as minimize contamination and drying time without increasing the energy (electricity) cost. Some solar is equipped with additional features to maintain the heat supply within the solar dryer during the slow drying period. The smoking process also has preferably been used by local and village people since firewood can be found easily elsewhere. However, smoking is suitable for certain foods such as meat, fish, or certain types of fruit such as banana and pineapples which particularly may enhance their flavor.

It is known that high temperature will reduce the moisture content faster, but it may affect the quality of dried material at surface. Drying at low temperature usually produces better products, but a long drying time is needed to get the desired end products. Therefore, it is important to study the behavior of drying for specific food material, and to determine the appropriate technique to get the final product quality required. Hence, here, study on the

behavior of drying for lemantak is needed to determine the best approach of drying technique, which lowers the moisture content as well as maintaining its quality.

1.2 Problem Statement

Traditionally processed sago starch, lemantak, has poor quality that are associated with moldy, smelly, water spot in the inner side of the packaging and pinkish or creamy color. Moldy and smelly starch may indicate the presence of bacterial activity, while the visibility of water spot may indicate incomplete drying, potentially leading to food spoilage. Hence, causes consumer rejection, market limitation and decreases in income for lemantak producers such as local farmers and small factories.

On the other side, commercial sago flour produced from modern factory has good appearance and quality that pass the requirement in Malaysia Standard for edible and non-edible starch as shown in Table 1.3 below.

Table 1.3: The requirement from Malaysia Standard for edible and non-edible starch (Karim et al., 2008)

Characteristics	Requirements	
	Edible starch	Non-edible starch
Moisture content	13% max	15% max
Color	90 min (‘L’ value)	0.4 red + 0.5 yellow (tentometer reading)
Peak viscosity (6% dry basis suspension)	600 BU	No data
pH of aqueous	4.5 – 6.5	4 min
Sulfur dioxide	30 ppm	No data
Particle size (through 125 μ m or 120 mesh size)	99% min	65% min
Crude fiber (dry basis)	0.1% max	1% max
Total ash (dry basis)	0.2% max	0.5% max

Table 1.3 shows the standard value required for starch trading and upstream industrial acceptance. As for example, lemantak need to have moisture content below 13% in dry basis; as well as whiteness index more than 90 in order meet the quality standard and to expand its market. A quick moisture content measurement on lemantak had indicated that it contains 20% moisture content in dry basis which is considered high.

Moisture content can be reduced through drying process. The drying process has been applied in lemantak production by using sun drying method. Sun drying is undoubtedly an economical method; however, lack of monitoring may lead to insufficient drying. Therefore, it is suggested to provide a reference for sago starch drying process that can be used not only for lemantak, but also for sago starch in general.

1.3 Gap of Research

Most of studies on sago starch are related to agronomy, genetic and molecular study, water, and waste management as well as starch modification, which focus more about encountering plantation problems, solving process hindrance, improving starch quality as well as altering starch properties. Traditional sago starch also had gained attention among starch researchers as several research had been conducted to improve its quality such as to optimize starch extraction (J. Moinsol, 2003; Pinyo et al., 2016), to improve sago processing (Mazlina Kamal et al., 2007), and the development of sago starch dryer (Pendita et al., 2014). Modification and treatment study combined with properties analysis is among research that has been applied to sago starch so far. For example, research conducted by Purwani & Thahir (2006), Yiu et al. (2008), and Lee et al. (2004); that have on effect of heat moisture treatment on sago noodle quality, effect of acid treatment on physicochemical properties and effect of photografting modification on sago starch flow behavior, respectively. The literature that is most related to this thesis is the one conducted by Maaruf et al. (2001) in which the effect of moisture content to sago starch gelatinization was studied. However, change in moisture content during drying was not assessed, as well as affected properties other than gelatinization. There are many studies that have been done to understand starch properties generally which will be discussed further in Chapter 2, but there are yet no studies about the

effect of drying process to the properties of sago starch. Similarly, there are many studies that have been done on drying kinetic of various starch types, but there are no studies of sago starch drying kinetic. This knowledge is very important because it will give idea on the inter-relation between various factors such as drying methods, moisture removal kinetics, drying parameters, drying variables (such as drying time, drying temperature, air velocity et cetera) with starch properties and quality.

1.4 Research Objective

The main objectives of the research are as follows:

1. To investigate the effect of drying conditions on the sago starch properties from different processing methods and different moisture content.
2. To determine the effect of drying parameter and methods on the final moisture content of sago starch.
3. To evaluate the effect of drying on the kinetic behavior and moisture diffusivities of sago starch.
4. To analyze the effect of drying temperature and method on the mathematical model of sago starch.

For the first objective, characterization was done for wet and dried sago starch. Wet sago starch is un-dried sago starch extracted in laboratory, while dried sago starch includes dried sago starch extracted in laboratory, lemantak and sago flour from modern factory. The properties analyzed were the sago starch composition, resistant starch content, granule morphology, granule size, starch viscosity, molecular changes, and thermal stability.

The second and third objective involve the analysis of final moisture content, drying kinetic and moisture diffusivities of sago starch as the effect of different variables and parameters such as drying temperature, air velocity, sample thickness, sample position and drying method.

The fourth objective used ten selected empirical models, a simplified Fickian model and the Characteristic Drying Curve model to search for drying model that can assist for sago starch drying prediction.

1.5 Research Questions

1. Does different starch processing method and final moisture content affect sago starch properties?
2. What is an optimum drying condition to have an ideal final moisture content of below 13% dry basis?
3. How does the kinetic behavior and moisture diffusivities change in corresponding to different drying conditions?
4. Which drying models can be used to analyze the drying kinetics of sago starch?

1.6 Hypothesis

1. Different starch processing methods affect sago starch properties while different final moisture content does not affect sago starch properties.
2. The optimum drying condition to have final moisture content below 13% dry basis is to dry sago starch at high temperature but below its gelatinization temperature, high air velocity and small amount in a batch.
3. The effect of temperature on drying lemantak can be more significant compared to the air velocity.

4. Empirical and simplified Fickian drying models can predict lemantak drying profiles better compared with the characteristics drying model.

1.7 Scope of Research and Its Limitations

The sago starch samples used in this study are lemantak and sago flour, which were bought from nearby shops in Kuching, and fresh sago, prepared in laboratory. Properties analysis involves proximate analysis such as moisture content, amylose content, protein, lipid and ash content. Besides, it also involves resistant starch analysis, granules morphological analysis, particle size, color, viscosity, FTIR spectrum analysis and thermo properties analysis. Drying methods comparison involves comparison on few types of drying methods such as continuous and intermittent oven drying, sun drying, microwave and freeze drying. The differences on EMC, drying kinetics and model fitting between each drying method were observed. Further drying experiments and analysis were done through convection oven drying by changing few variables such as temperature within range of 50 to 80°C, air velocity within range of 0 to 0.965 m/s, sample thickness within range of 0.5 to 2 cm, and sample position in oven (middle-top, middle down and side). The drying models assessed were thin layer models, simplified Fickian model and Characteristic Drying Curve model.

1.8 Research Novelty

The outcomes of this study contribute to the fundamental knowledge on the effect of processing methods, especially the drying process to the properties of sago starch. Most studies on sago starch found in the literature are related to agronomy, genetics, molecular study, starch modification and waste management. Hence, the research novelty of this work relates to the analysis on the properties of sago starch and its drying kinetics. Sago starch

used in this work was from the sago palm trees that are grown in Sarawak, which has not been studied in detail. Properties analyzed were the composition, morphology, particle size, color, viscosity, molecular structure, and thermal stability. The effect of heating or drying on the properties of sago starch has also been included. Study on the effect of drying was extended to cover the drying kinetics for sago starch, which has not been discussed widely in the literature. In this work, the effect of drying temperature, air velocity, sample thickness, sample position, and drying method on the EMC, drying profiles and moisture diffusivities were assessed. The relationship between EMC with these variables was determined. The suitability of empirical models, Fickian Diffusion model and Characteristic Drying Curve to assess the drying kinetics of sago starch was also studied, and all the fitted drying model parameters were estimated.

CHAPTER 2

LITERATURE REVIEW

This chapter reviews the properties and kinetic study of dried sago starch. Firstly, the sago palm and its starch are introduced. Then several analysis suitable for properties investigation is introduced, as well as the effects of drying to the starch properties that are found in literature is elaborated. The effect of drying to starch kinetic is mentioned in the next subtopic. The application of drying model is introduced, together with the elaboration on four types of drying models that may be suitable for sago starch drying kinetic. Lastly, the literature review is summarized.

2.1 Sago Palm and Its Significance

Sago palm is a kind of big tropical plant with huge trunks up to 40 cm diameter and height up to 25 m. It has pinnate leaves that look like a crown as in bundle, which can grow as big as 9 meter long. There is many type of sago palm but specifically, but the scientific name for sago palm used in this study is called the *Metroxylon sagu*. Sago palm lives well in peat soil but is also able to survive in hard soil conditions such as swampy, flood or acidic soil. The mature palm grows new plants as a sucker which eventually makes a cluster of sago palm around the original mother as can be seen in Figure 2.1.



Figure 2.1: Sago Palm Plantation Area in Mukah, Sarawak (Whye Kit et. al., 2019)

Sago palm accumulates its starch inside the trunk and underneath the pith from base-upward. The starch is best to be harvested around 9 to 12 years after planting. Therefore, sago palm possesses the starch with the longest harvest time among other starch crops (Whye Kit et al., 2019). However, this disadvantage is outweighed by its very high productivity of 250 kg of sago starch per palm as well as very high toleration of soil condition, which can tolerate wet-swampy peat soil and even flood condition (Whye Kit & Chung, 2020). Moreover, the yield of starch from sago palm was reported to be the highest among other starches which is about 3 to 4 times higher than rice, corn or wheat, and about 17 times higher than cassava (Shamini, 2020). In these recent years, sago palm has been credited by researchers as the ‘key starch crop of the 21st century’ and ‘crop par excellent’ (Whye Kit et al., 2019). Sago palm is one of the oldest tropical plant exploited by human specifically for its starch as a carbohydrate source (Whye Kit & Chung, 2020).

Nowadays, sago palm is strategically planted in most of ASEAN countries such as Malaysia, Indonesia, Thailand, and Philippines. As for Malaysia, sago palm has become an important agricultural commodity in Sarawak, since there are about 1.69 million hectares of peat soil area which have been considered for sago palm plantation (Whye Kit et. al., 2019). Recently, there are 21 modern sago processing plants all around Sarawak. On 2022 February 21st, The Star has reported that Sarawak has become one of the world largest sago starch producer which had produced as much as 37,884 tons of sago starch which was worth RM78.3 million, as of 2021. Sarawak has the potential to focus on sago industry since sago palm can grow well in peat soil which also covers 75% of total land area in Sarawak. Furthermore, sago palm can thrive in harsh swampy peat environment which covers an area of 1.5 million ha that is equal to 12% of area in Sarawak (Whye Kit et. al., 2019)).

2.2 Sago Starch and Its Beneficial

Sago starch contains mainly carbohydrates that are composed from amylose and amylopectin as shown in Figure 2.1. The basic structure of amylose and amylopectin is the molecules of glucose that are bonded together to form a long chain. However, the glucose molecules in amylose are linear while in amylopectin are branching as can be seen also in Figure 2.2.

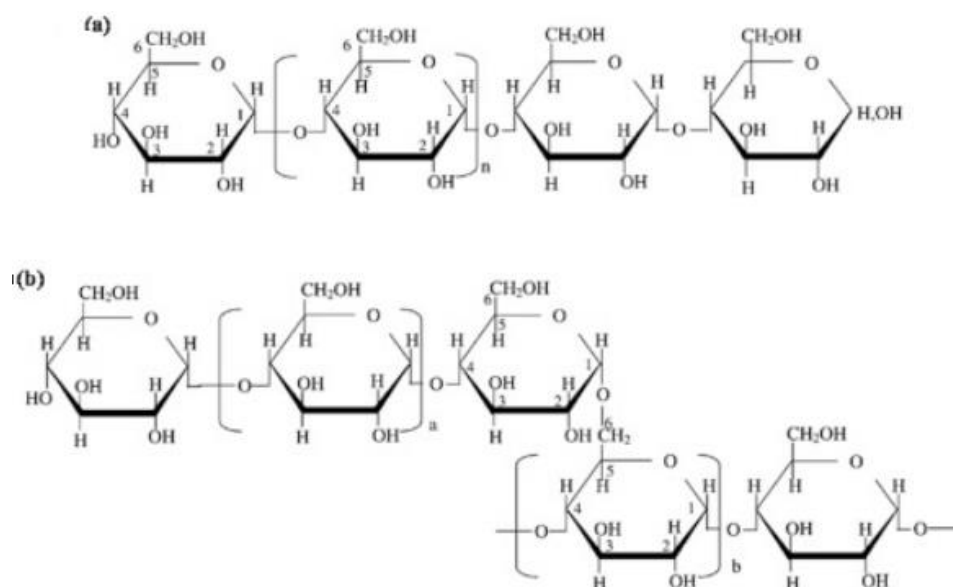


Figure 2.2: Structure of amylose, (a) and amylopectin (b) in starch (Tester et al., 2004)

Sago benefits in its compositions. It has a high carbohydrate content of 84.7g per 100g, which is higher than rice that is 80g per 100g, and wheat flour of 77.3g per 100g. The Ministry of Agriculture of Sarawak had stated that sago as a local food has a great potential to be developed in supporting food diversification because it contains high carbohydrate. As for consumers, sago has the effect of being filling at a lower caloric content, which is good for diet. Its protein content is very low and free from casein and gluten protein (Andi Patiware et al., 2016). Therefore, it is good to be consumed by people with gluten intolerance disease. In addition, sago is safe to be consumed by diabetic mellitus people as it does not raise blood glucose levels immediately (low glycemic index). Sago also contains high amount of resistant starch which can prevent the risk of constipation and colon cancer (Karim et al, 2008). Last but not least, Dewayani (2022) had suggested sago starch to be the main staple food by replacing rice and cassava for its carbohydrate and caloric contents.

In the early days, sago starch was consumed as a staple food for its carbohydrate as the source of energy. It was also used as the main ingredients to make some local delicacies and traditional cookies. Sago starch has intrinsic taste and behavior to prepare certain foods which make it favorable among local people. For instance, when lemantak is mixed with hot water, it produces different viscosity and color better than commercial sago flour, that is linut (as mentioned in Chapter 1).

Nowadays, sago starch is used in many food industries as a basic ingredients to produce noodles, vermicelli, sago pearls, bread, cakes, and cookies (Zhu, 2019). It also has been used as a thickening agent in soup, gravy, creamer, and jam (Zhu, 2019). Recently, demand for sago starch in non-food industries has increased in which its functional properties have been used applied in making supplements, adhesive, bio-degradable plastic, alcohol and cosmetics (P. Ghalambor, 2022). Furthermore, it is predicted that the demand of sago starch will increase in the future as it is inexpensive and has low processing cost (Zhu, 2019).

2.3 Analysis on Starch Properties

Analysis on starch properties involves physicochemical properties analysis and functional group analysis. Starch properties have been studied by many researchers in many research area such as for characterization (Contreras-Jimenez, 2019), post-treatment effect (Donmez, 2021), and variation comparison (Baini et al., 2017). Physicochemical analysis involves composition analysis, functional properties analysis, physical properties analysis and thermal properties analysis. Functional group properties of starch focus on the molecular aspect which is affected by the interaction between amylose and / or amylopectin with other

molecules such as water, fat, and protein. The list of previous research findings on starch properties analysis is concluded in Table 2.1 below.

Table 2.1: Previous research findings on starch physicochemical properties

Properties	Analysis/Equipment	Findings	Reference
Composition	AOAC method	Moisture content, fat/lipid, protein, and ash content for three sago starch types	Santoso et al., 2021
		Moisture content, fat/lipid, protein and ash content for sago starch, corn, and potato	Du et al., 2020
Starch content	Luff Schroll method (SNI 2011)	Starch content for three types of sago	Santoso et al., 2021
	Megazyme assay kit	Starch content of sago starch, corn, and potato	Du et al., 2020
Amylose content	Gujral and Rosell (2004) method	Amylose and amylopectin content for three types of sago	Santoso et al., 2021

	Li and Zhu method	Starch content of sago starch, corn, and potato	Du et al., 2020
Resistant starch (RS) content	Wicklow assay kit	RS content of sago starch, corn, and potato	Du et al., 2020
	AOAC 2002 and AACC 38-40 method using assay kit	RS content of lemantak	Arshad et al., 2018
Granules morphology	Scanning electron microscope (SEM)	SEM micrograph of native and dual modified sago starch.	Ghalambor et al., 2022
		Sago starch granules shape comparison between native and oxidized.	Sumardiono et al., 2021
Granule size	Particle size analyzer	Granules shapes of sago starch, corn, and potato	Du et al., 2020
		Granules size distribution of sago starch, corn, and potato	Du et al., 2020

Color	Calorimeter	Color of starch isolated from chestnuts	Guo et al., 2019
	Chroma meter	Color of potatoes starch varieties	Wang et al., 2020
Pasting properties	Rapid Visco Analyzer	Amilograph characteristic of three sago starch types	Santoso et al., 2021
		Peak viscosity reduction in treated sago starch.	Hartiningsih et al., 2020
		Correlation between Peak Viscosity (PV) and amylose content.	Du et al., 2020
		Change on gelatinization and enthalpy temperature due to gamma irradiation	Lee et al., 2021
Thermal properties	Differential Scanning Calorimetry (DSC)	High gelatinization temperature and low gelatinization enthalpy in	Du et al., 2020

		sago starch compared to corn and potato. Consistent finding with pasting properties.	
	Thermogravimetric (TGA)	Thermal decomposition of sweet potato starch	Liu et al., 2019
Functional group	Fourier Transform Infrared (FTIR) spectroscopy	Confirmation on glycosidic breakdown	Lee et al., 2021
		Comparison of functional group absorption between native and oxidized sago starch.	Sumardiono et al., 2021
		Alteration possibility in amorphous and crystalline regions in modified sago starch. Molecular bond losses in modified sago starch.	Hartiningsih et al., 2020

		Functional group comparison between sago starch, potato and corn. The degree of order for sago, potato and corn starch structure.	Du et al. 2020
		New peaks observed in dual modified sago starch	Dewi et al., 2022

In composition analysis, the starch composition is determined in percentage with the total of 100 percent. Moisture content is one of the main items to be determined in the composition analysis. It presents the amount of water that exists in the material after dried thoroughly. In natural states, sago starch granules are binding with water molecules. The amount of water molecules attached is called moisture content. Composition analysis also includes analysis on fat, protein, and ash content. These three items are considered as contaminants that usually exist in a very small percentage but can affect some of the reactions that occurred in the starch. Moisture content, protein, lipid, and ash content are usually measured in accordance with the AOAC official methods (Du et al., 2020).

Starch content is determined by referring to the total of amylose and amylopectin content. Some researchers go into more detail by determining each of amylose and amylopectin content (Du et al., 2020). However, the focus is more towards the amylose content as it is more favorable as amylose plays a major role in viscosity. Total starch content,

amylose and amylopectin content can be analyzed by using Megazyme assay kit as (Du et al., 2020). Some researchers such as Du et al. (2020) also analyzed resistant starch content since it has gain attention among food scientist in recent years. Resistant starch content is measured by using resistant starch assay kit in as stated in the method of AOAC 2002 (Arshad et al., 2018).

Starch morphology has been analyzed intensively by researchers by using Scanning Electron Microscope (SEM) in which the granules can be observed under up to 2000 times of magnification or in other words, micro-object can be seen clearly (Hartiningsih et al., 2020; Dewi et al., 2022; Ghalambor et al., 2022).

Granule size can influence starch textures and has been studied by many researchers. Hartiningsih et al. (2020) not only used SEM to analyze granule`s morphology, but also to measure the granule`s size. However, most of the researchers used Particle Size Analyzer as it can give the overall granule size distribution so that the average size is more precise. For instances, Du et al. (2020) used particle size analyzer to compare granule size between two types of sago starch together with potato and corn starch granule size. Du et al., 2020 also mentioned that granule size is influenced by many factors such as the botanical origin, starch extraction method and molecular structure. Granule size also may affect their physicochemical properties and enzymatic digestion.

Starch also can vary in certain range of whitish color as the effect of sago palm type, as well as sago starch extraction and process (Wang et al., 2020). Drying also can change the color of sago starch as found by Guiné (2018). Therefore, color measurement is also an important aspect in starch properties analysis. Chroma meter and calorimeter are usually

used to analyze starch color as it uses the L*a*b standard color system to differentiate color (Guo et al., 2019; Wang et al., 2020).

Analysis on viscosity is an important aspect in characterizing starch as it will give insight regarding its function as an ingredient used in downstream food industries (Tabilo-Munizaga & Barbosa-Canovas, 2005). In wood industry, starch is always used as an adhesive extender or even the adhesive itself, in which its viscosity play a major role in determining the glue bonding (Yu et al., 2015; Sumadiwangsa, 1995). Viscosity has been studied intensively by using Rapid Visco Analyzer as it is one of the most desired properties of sago starch (Ariyantoro et al., 2018). Different type of sago starch has different viscosity (Sarungallo et al., 2021) and native sago starch has different viscosity from modified sago starch (Hartiningsih et al., 2020). Modified starch showed different viscosity as found by Lee et al. (2021) when treated sago starch with gamma light.

Studying thermal properties is also important to understand the thermal stability, decomposition, and degradation of starch towards heat. Starch thermal behavior had been studied by Du et al. (2020) and Cia et al. (2019) through Differential Scanning Calorimetry (DSC) and Thermogravimetric Analyzer (TGA), respectively. DSC and TGA have been widely used to analyze the thermal properties of sago starch. DSC can provide energy changes within cycles while TGA can provide mass changes while heating at a constant rate or constant temperature over time.

Finally, functional group analysis is important to identify functional group changes that could lead to better understanding of the functional properties analysis. The Sago starch molecule can be studied through Fourier Transform Infrared Spectroscopy (FTIR) analysis as the different IR light is absorbed by different chemical bonds, and the

light spectra is presented as the results. The subjection of sago starch into FTIR will generate a spectra fingerprint with wavelength that range around 800 to 3600 cm^{-1} . Different starches have different FTIR spectra. Moreover, modified starch should also has different spectra than its native. Zailani et al. (2022) and Hartiningsih et al. (2020) are among the researchers that had successfully used FTIR to identify the changes in sago starch composition in term of chemical bond. Generally, Fourier transform infrared (FT-IR) spectroscopy has recently been applied as part of starch properties analysis as it allowed rapid molecular characterization (Du et al.,(2020)) and biochemical changes identification for treated samples (Hartiningsih et al., (2020); Lee et al., (2021); Sumardiono et al., (2021); Dewi et al., (2022)).

2.4 Effect of Drying on Starch Properties

Moisture content is defined as the amount of water present in moist material. In starch, there are two types of water states that contribute to moisture content namely bound water and free water. Bound water is already attached to starch molecules since it is in the palm state, while free water was obtained during extraction process (Zhiqiang et al., 1999). Free water can be removed through drying process to reduce the amount of moisture content. As for sago starch, the quantity of bound water is influenced by the sago palm harvesting stage as discovered by Moinsol (2013) that younger palm low starch content with higher moisture content, while matured palm has high starch content and lower moisture content.

Some researchers have also reported that moisture content is one of the key aspects that influences starch properties. For instance, the mechanical properties that have been studied by Farid et al. (2021), the structural changes (Su et al., 2022), gelatinization properties (Li et al., 2022) as well as retrogradation (Ding et al., 2019).

Drying process is subjected to starch to reduce its moisture content. Drying is also important to reduce bacterial growth risk, expanding shelf life, promoting export market, reduce weight for packaging and delivery, and improve starch quality such as color and general appearance. Each starch has a different maximum moisture content to avoid microbial activity that can lead to food spoilage. For instance, fungal growths were found to be 13.0% for rice grains, 12.9% for glutinous rice grains, 9.6% for rice flour, 10.6% for glutinous rice flour, 10.7% for wheat flour and 10.5% for corn flour (Abdullah et al., 2000). The maximum moisture content for edible and non-edible sago starch as stated in Malaysia Standard (Table 1.3) are 13% and 15%, respectively.

However, drying also can cause properties to change including improvement or degradation. In food, properties change could be such as nutritional degradation or physicochemical properties change. The information on drying characteristics and properties changes will give insight for further optimization of the drying process (Guiné, 2018). This is because the interaction of water with starch is related to several important phenomenon such as swelling, melting, pasting, gelatinization and retrogradation which are widely and intensively applied in process industry. There are also several properties that might be affected by drying process such as color, morphology, granule size, structure, functional and thermal properties.

Drying may change starch color to either darker or whiter. Timm et al. (2020) found that starch from some corn genotypes becomes more greenish and yellowish, due to increase in pigmentation, as the drying temperature increases from 30 to 90°C. Starch also can become darker due to Maillard Reaction (Khoozaini et al. 2019). Timm et al. (2020) also found that peak viscosity decreases as the drying temperature increases. While Khoozaini et al. (2019) also found that oven and freeze-drying increase the amylose and resistant starch

content, as for banana flour. In terms of size, Caparanga et al. (2017) found that higher drying temperature produces smaller granules, while higher drying air velocity produces bigger granules. Different drying methods also give different changes as found by Zhu (2018) in which oven and freeze drying resulted in different effects on starch surface. Moreover, starch structure also may change due to drying as found by He (2021) that hot air and freeze drying had destroyed the ordered structure of canistel starch.

2.5 Effect of Drying on Starch Drying Characteristics

Drying characteristics are related to the physical (mass) and mathematical aspect of drying process which provide information on drying kinetic, moisture diffusivity and activation energy, as well as enable the development of drying model. Study on drying kinetics of sago starch was not found in the literature. Hence, limited in-depth analysis on the relationship between the heating effect and the properties of sago starch. Drying kinetics of food found in the literature are mostly related to fruits such as banana, apples, onion, mushroom, and some types of starch, mostly wheat, corn, potato, and rice flour. Drying kinetics literatures found relates to the effect of time and temperature on the moisture content as well as nutrient content of food. Extended study on drying kinetics covers the use of models to study the behavior of drying.

Drying kinetics can be studied based on experimental data and predicted data from drying models. Drying kinetics of sago starch can be modeled empirically using first-order reaction kinetics or Characteristic Drying Curve, and mechanistically by using Fick's' Law of Diffusion as has been used by Baini and Langrish (2009). Empirical models include lumped-parameter models that generally predict only the average moisture content as a function of drying time, while Fickian Diffusion includes the diffusion coefficient which

able to predict moisture contents that varies with time and space. The concept of Characteristic Drying Curve assumes that the shape of the falling-rate drying curve, during hindered drying, is unique for each material. Fickian Diffusion can be used to describe moisture transport physically within material and gives results of more fundamental value compared with empirical models. However, empirical models are simpler as compared to the diffusion model. Due to this simplicity, they are sometime well accepted and applied in the study of fruit drying.

2.6 Drying Models for Starch

Drying models chosen for this work are empirical models, Fickian Diffusion model and Characteristic Drying Curve, which are commonly used to study the drying kinetics for food, including for fruits, vegetables etc. These models were chosen because it is suitable to demonstrate the exponential decrease of moisture content similar to the food drying phenomenon. The moisture content of drying food decreases with time, and it can be predicted using exponential expression well. Most empirical drying models predict the moisture content as a function of time using exponential expression. Simplified Fickian Diffusion model predicts the exponential decrease in moisture content as a function of time, incorporating the temperature and diffusivity. The drying characteristic model illustrates the drying behavior in terms of drying rate against the moisture content and consists of the constant and falling drying rate. This model has also been used for food drying.

2.6.1 Empirical models

The empirical model commonly used to evaluate the drying kinetics of food (Dikbasan, 2007) is showed as in Equation 2.1 below.

$$MR = \frac{M_t - M_e}{M_i - M_e} \quad \text{Equation 2.1}$$

The drying kinetics of fruits (Kiranoudis et al., 1997) and vegetables (Krokida et al., 2003) has been empirically predicted using a first-order approach, as described by Equation 2.2.

$$-\frac{dX}{dt} = k_d (X - X_e) \quad \text{Equation 2.3}$$

This model has been found to fit the experimental moisture content for materials that were dried continuously very well (Kiranoudis et al., 1997; Maskan, 2000; Phoungchandang & Woods, 2000; Baini & Langrish, 2004; Hofsetz & Lopes, 2004; Sousa & Marsaioli, 2004). The model was first suggested by Lewis (1921). Here, the drying rate is assumed to be proportional to the moisture content difference with k_d as the drying constant. The equation may be integrated to give the following ones:

$$MR = \frac{M - M_e}{M_i - M_e} = \exp(-k_d t) \quad \text{Equation 2.4}$$

$$X = (X_o - X_e) \exp(-k_d t) + X_e \quad \text{Equation 2.5}$$

These equations express first-order kinetics in terms of the moisture ratio (Equation 2.4) to give a predicted content or fitted moisture content, X , (Equation 2.5) that decreases exponentially with time. X_e is the equilibrium moisture content expressed on a dry basis. There are other expressions for the moisture ratio (MR) that have been used to describe the

drying kinetics for fruits. Togrul and Pehlivan (2004) used the models in Table 2.2 in their drying work for grapes, peaches, figs, and plums. These models have been reported in literature and are frequently used to fit empirical correlations for describing behavior of natural products.

Table 2.2: Empirical models for fruit drying, Togrul and Pehlivan (2004)

Model and references	Model equation	Equation
Newton O'Callaghan et al. (1971)	$MR = \exp(-kt)$	Equation 2.6
Page Page (1949)	$MR = \exp(-kt^n)$	Equation 2.7
Henderson and Pabis Henderson and Pabis (1969)	$MR = a \exp(-kt)$	Equation 2.8
Logarithmic Yagcioglu et al. (1999)	$MR = a \exp(-kt) + c$	Equation 2.9
Two term exponential Sharaf-Eldeen et al. (1980)	$MR = a \exp(-k_0 t) + b \exp(-k_1 t)$	Equation 2.10
Wang and Singh Wang and Sing (1978)	$MR = 1 + at + bt^2$	Equation 2.11
“Approximation of diffusion” Sharaf-Eldeen et al. (1979)	$MR = a \exp(-kt) + (1-a) \exp(-kbt)$	Equation 2.12

Verma Verma et al. (1985)	$MR = a \exp(-kt) + (1-a) \exp(-gt)$	Equation 2.13
Modified Henderson and Pabis Karathanos (1999)	$MR = a \exp(-kt) + b \exp(-gt) + c \exp(-ht)$	Equation 2.14

They found that the models above were the best ones to describe the drying of the fruits in question; the “approximation of diffusion model” for apricots (non-pre-treated or SO₂–sulphured) and figs; the modified Henderson and Pabis model for apricots (NaHSO₃–sulphured), grapes and plums; and the Verma model for peaches. These results showed that the best empirical model depends on the material, and that there may be more than one empirical model which is reasonable for each type of fruit. The non-uniqueness of the ‘best’ empirical model is a significant source of uncertainty in the use of empirical models. In their work, two different models were found to be reasonable for apricots that were treated by different chemicals (SO₂ and NaHSO₃ were used in this work).

The drying constant has been correlated with the air-drying conditions and the material dimensions by Kiranoudis et al. (1997), as shown in Equation 2.15 and it has also been associated with the temperature using the Arrhenius relationship by Phoungchandang and Woods (2001), as shown in Equation 2.16.

$$k_d = k_0 d_p^{k_1} T^{k_2} u^{k_3} a_w^{k_4} \quad \text{Equation 2.15}$$

$$k_d = k_{dr} \exp\left(-\frac{D_{EE}}{T}\right) \quad \text{Equation 2.16}$$

The drying constant has units of time^{-1} . Here, d_p is the diameter of the dried material, T is the drying temperature, u is the air velocity and a_w is the relative humidity.

The equilibrium moisture content, X_e , is sometimes treated as zero to simplify the mathematical solution (Sousa and Marsaioli, 2004). In reality, it is significantly greater than zero and is a function of the relative humidity and the temperature (Kiranoudis et al., 1997).

In the work of Dandamrongrak et al. (2002), the moisture ratio has been applied for drying bananas using three models as shown in Table 2.3. In their work, bananas were treated using four different pre-treatments (blanching, chilling, freezing, and freezing combined with blanching) prior to drying. The drying conditions were a temperature of 50°C , an air velocity of 3 ms^{-1} and relative humidity of 10 – 35%.

Table 2.3: Empirical models for banana drying (Dandamrongrak et al., 2002)

Model name	Model equation	Instantaneous moisture content	Equation number
Newton	$MR = A_D \exp(-k_{ad}t)$	$X = X_e + A_{D1} \exp(-k_{ad}t)$	Equation 2.17
Two-term	$MR = A_D \exp(-k_{ad1}t) + B_D \exp(-k_{ad2}t)$	$X = X_e + A_{D1} \exp(-k_{ad1}t) + A_{D2} \exp(-k_{ad2}t)$	Equation 2.18
Page	$MR = A_D \exp(-k_{ad}t^{ND})$	$X = X_e + A_{D3} \exp(-k_{ad}t^{ND})$	Equation 2.19

When these empirical models are typically applied, the radius of the banana and the diffusion coefficient are constant so the real effects of shrinkage and the dependency of the diffusion coefficient on the temperature and other factors are typically ignored. The fitting of the average moisture contents using the models was found to be satisfactory. The fitted equilibrium moisture contents (kg per kg dry basis) were found to be in the range of (0.221

– 0.443) for the Newton model, (0.072 – 0.158) for the two-term model and (– 0.053 – 0.045) for the Page model. Based on these results, the two-term model was chosen as the best model to represent the drying of bananas.

The analytical solution of the diffusion equation was also used by Nogueira and Park (1992). They fitted the average moisture contents for whole peeled bananas that were dried in a range of temperature from 50 -70°C and air velocities from 0.5 – 1.5 m/s. They reported activation energies for banana that were from 15.5 to 25.3 kJ/mol, while the diffusion coefficients were from 2.6×10^{-10} to 6.5×10^{-10} m²/s. However, these values are questionable, because the use of the analytical solution of the diffusion equation by them is suspect, due to the unphysical assumptions made in this solution about the diffusion coefficient being constant for each experiment and about shrinkage being ignored.

2.6.2 Fickian Diffusion Models

A diffusion model based on Fick’s Laws of diffusion is a model, which has frequently been used in literature to describe the diffusion of moisture in dried fruits (Baini & Langrish, 2008). For axially symmetric cylindrical coordinates, an unsteady-state mass balance can be expressed as shown in Equation 2.20.

$$\frac{dX}{dt} = \frac{1}{r} \frac{d}{dr}(rJ) \quad \text{Equation 2.20}$$

During drying, moisture moves through passageways, which can be in any direction, longitudinally or laterally from the core to the surface of the bananas. The diffusion path has often been described to be in radius direction as for round object, and the lateral direction, as for a cylindrical shape of material. For example, a whole banana with a cylindrical shape, the change in length was found to be small compared with the change in radius (Baini &

Langrish, 2007). Therefore, here, the path length, r , in Equation 2.20 is the radius of the banana.

Moisture diffusion in drying fruits may be described as driven by the differences in the chemical potentials of moisture within the fruit. At equilibrium, these chemical potentials, μ_J , are the same, but any changes in the surface conditions will result in a difference in chemical potential. The moisture flux, J , defined as the moisture flux between cylindrical elements of the drying material (banana) may be associated with a function of the chemical potential gradient across the path length where the diffusion occurs, as illustrated by Equation 2.21. A similar approach has been used for describing diffusion in drying timber. The following equations are taken directly from Keey *et al.* (2000):

$$J = -B_J X_J \frac{d\mu_J}{dr} \quad \text{Equation 2.21}$$

In Equation 2.21, B_J is a coefficient. The moisture flux can also be expressed in terms of the moisture content difference as shown in Equations 2.22 and 2.23.

$$J = - \left[B_J RT \frac{d \ln a_w}{d \ln X_J} \right] \frac{dX_J}{dr} \quad \text{Equation 2.22}$$

$$J = -(D) \frac{d(\rho_b X)}{dr} \quad \text{Equation 2.23}$$

In Equation 2.22, the moisture flux is expressed in terms of water activity, a_w , and moisture concentration, X_J . The moisture concentration (kg/m^3) is the product of the moisture content, (kg/kg) and the oven-dry density of the material, ρ_b (kg/m^3), which gives a relationship between the moisture flux and the moisture content as shown in Equation 2.23. The term D , in the brackets in Equation 2.23 is called the moisture diffusivity.

The moisture flux, as defined in Equation 2.21, may then be substituted into Equation 2.20 to give a diffusion model for cylindrical coordinates as shown in Equation 2.24.

$$\frac{dX}{dt} = \frac{1}{r} \frac{d}{dr} \left[r \frac{dDX}{dr} \right] \quad \text{Equation 2.24}$$

The diffusion model illustrated by Equation 2.24 can be solved analytically (Crank, 1975) using the initial and boundary conditions, as illustrated by Equations 2.25 to 2.27, to give a solution as given in Equation 2.28.

$$t = 0, \quad X = X_0, \quad 0 \leq r \leq r_h \quad \text{Equation 2.25}$$

$$t > 0, \quad r = r_b, \quad X = X_e \quad \text{Equation 2.26}$$

$$r = 0, \quad \frac{dX}{dr} = 0 \quad \text{Equation 2.27}$$

$$MR = \sum_{n=1}^{\infty} \frac{4}{\beta_n^2} \exp \left[-\frac{\beta_n^2 Dt}{r^2} \right] \quad \text{Equation 2.28}$$

Equation 2.28 can be simplified by considering the first term only and may be expressed as shown in Equation 2.29.

$$MR = \frac{4}{\beta^2} \exp \left[-\frac{\beta^2 Dt}{r^2} \right] \quad \text{Equation 2.29}$$

MR in Equation 2.28 and 2.29 is the moisture ratio, as illustrated in Equation 2.30.

$$MR = \frac{X - X_e}{X_0 - X_e} \quad \text{Equation 2.30}$$

Equation 2.29 has also been expressed using different terms (Akpınar & Dincer, 2005; Dandamrongrak et al., 2002), such as those shown in Equations 2.31 to 2.34.

$$MR = G \exp(-S_t) = A_1 B_1 \quad \text{Equation 2.31}$$

$$A_1 = G = \frac{4}{\beta^2} \quad \text{Equation 2.32}$$

$$S = \frac{\beta^2 D}{r^2} \quad \text{Equation 2.33}$$

$$D = \frac{S r^2}{\beta^2} \quad \text{Equation 2.34}$$

In the numerical solution of the diffusion model, the banana is divided into several finite volumes, and the properties – moisture content, temperature, and diffusivity – in each finite volume are estimated. In contrast to the numerical solution of the diffusion model, the analytical solution such as that expressed in Equations 2.28 to 2.29 (simplified Fickian model) often assumes that the moisture content within the material is initially uniform and equal to the initial moisture content, that the diffusion coefficient is constant, and that the surface of the material instantly comes to equilibrium during drying. The external conditions, and hence the equilibrium moisture content, are assumed not to change during drying, which may make the analytical solution of the diffusion models less appropriate than the numerical solutions for intermittent drying. The characteristic length, r , is sometimes assumed to be constant in the analytical solution of the diffusion model, and this is usually not true because foods shrink during drying. In addition, the simple Fickian diffusion model is able to predict diffusion for condition two or less component such as water and material only (Haart & Spiller, 2009).

2.6.3 Characteristic Drying Curve

The characteristic drying curve is an analysis to characterize the shape of falling/hindered drying curve in a material that had shown the falling curve. The concept of a characteristic drying curve suggests that there is a unique shape of the falling or hindered drying curve for a particular material. The shape does not change significantly with different drying conditions over some range of conditions. This concept is discussed in more detail in Keey (1992). A characteristic curve of a moist material might be drawn up from a single experimental rate-drying curve. The relative drying rate is defined as in Equation 2.35.

$$R_{DR} = \frac{N}{N_v} = f(\Phi) \quad \text{Equation 2.35}$$

N_v is the unhindered drying rate. It may be taken as the maximum rate if there is no clear unhindered drying period. In Equation 2.36, Φ is the characteristic moisture content, which is the ratio of the free moisture content ($X - X_e$) to the moisture content difference between the critical point and the equilibrium moisture content.

$$\Phi = \frac{X - X_e}{X_{cr} - X_e} \quad \text{Equation 2.36}$$

X is the volume-averaged moisture content, X_{cr} is the moisture content at the critical point and X_e is that at equilibrium. For products with no clear first drying phase, the critical moisture content is usually assumed to be equal to the initial moisture content (Jannot et al., 2004).

The plot of the relative drying rate against the characteristic moisture content is normalized to pass through the point (1, 1) at the critical point and to pass through the point (0, 0) at equilibrium. The critical point is the point of transition from the unhindered drying

rate (or the maximum drying rate if the unhindered period is unclear) to the hindered drying period. This approach has been used by Jannot et al. (2004) to describe the drying of bananas. They observed that there were two significant drying phases. The first phase was an exponential decrease of the drying rate for the characteristic moisture content from ϕ_2 to 1, as described by Equation 2.37. Then, it was followed by a linear period of drying for the characteristic moisture content from ϕ_1 to ϕ_2 , as described by Equation 2.38. The drying rate, as a function of the characteristic moisture content in the range from ϕ_1 to 0, was not described mathematically by Jannot *et al.* (2004), and therefore it is not included here. The values of the characteristic moisture content, ϕ_1 and ϕ_2 , which correspond to transitions between the drying periods, were determined graphically to be 0.04 and 0.2, respectively.

$$f(\phi) = a_J \exp(b_J \phi) \text{ for } \phi_2 < \phi < 1 \quad \text{Equation 2.37}$$

$$f(\phi) = c_J \phi + d_J \text{ for } \phi_1 < \phi < \phi_2 \quad \text{Equation 2.38}$$

They also suggested correlations for the moisture content as a function of time by solving Equations 2.37 to Equation 2.38. These correlations are presented in Equations 2.39 and 2.40. The fitted parameters were given as $a_J = 0.079$, $b_J = 2.541$, $c_J = 0.478$, $d_J = -0.004$ and $N_v = 1.716 \text{ kgkg}^{-1}\text{s}^{-1}$.

$$X(t) = X_e - \frac{1}{b_J} (X_0 - X_e) \ln \left[\exp(-b_J \phi) + \frac{a_J b_J N_v}{X_0 - X_e} t \right] \text{ for } \phi_2 < \phi < 1 \quad \text{Equation 2.3}$$

$$X(t) = X_e + (X_0 - X_e) \left(\phi + \frac{d_J}{c_J} \right) \exp \left[-\frac{c_J N_v}{X_0 - X_e} (t - t_2) \right] - \frac{c_J}{c_J} \text{ for } \phi_1 < \phi < \phi_2 \quad \text{Equation 2.40}$$

This concept of using a characteristic drying curve implies that the ratio of the drying rate relative to the unhindered rate at given moisture content is independent of the external drying conditions.

2.6.4 Models for Estimating the Diffusivity

Unlike the first order empirical models and characteristic drying curve models, diffusion models include the diffusion coefficient, which reflects a possible physical phenomenon that may occur during drying. Diffusion has often been described as one of the transport mechanisms for moisture transfer during drying (Aguerre & Suarez, 2004), and the effect of temperature is incorporated, as illustrated in Equation 2.41.

$$D = D_r \exp \left[-\frac{D_{EF}}{(T+273)} \right] \quad \text{Equation 2.41}$$

Here, D_r is the pre-exponential factor and D_{EF} is the activation energy.

The effective moisture diffusivity was calculated using the method of slopes. When logarithm of MR values vs drying time was plotted in accordance with Equation 3.5, straight lines were obtained at all temperatures and sample thickness investigated. Linear regression analysis was employed to obtain values of diffusion coefficients for different drying conditions from the slope of the straight lines obtained. Several researchers had successfully measured the moisture diffusivity of starch such as Toğrul and Toğrul (2007); Sobukola et al. (2008); Benali (2012); Aregbesola et al. (2015) and Boutelba et al. (2018).

2.7 Summary of Literature Review and Research Gap

Review on food drying shown that drying starch can be done through several methods but the most such as sun drying, oven drying, microwave and freeze drying. However, considering the background of the study which is to give light on community-based sago starch processing, which also considered on other factors such as affordability, hygienist and economical process, oven drying gives the best promises to local farmer and small medium enterprises industries. Besides, intermittent oven drying also can be a good alternative to meet the above concern. According to several studies, the temperatures of 40°C to 80°C are commonly used to dry food including starch. Higher temperatures are not necessary as it leads to starch damage. Air velocity, thickness, and position during drying also have been found to improve drying process in term of drying rate and kinetic. Besides, study on drying process has been correlated with its effect towards dried material which focus on the physical alteration, composition changes or functional properties differences. Therefore, this work will present the comparison of not only on the composition but also on the physicochemical and functional properties of modern and traditionally processed sago starch as there have different drying process. Besides, drying model that fit sago starch drying kinetic is still unknown, thus, the information will be useful for future reference. The selected drying models are the ten most used model for foods and fruits. Besides the empirical model, information gained from Fickian model also beneficial due to its simplicity and mathematical tractability.

CHAPTER 3

METHODOLOGY

In this chapter, the methods used to conduct this study are presented. The methods can be divided into three sections which are characterization of sago starch, drying experiment to study the effect of heating on the properties of sago starch and drying analysis that includes study on drying kinetics for sago starch. First, the procedure for sample preparation is introduced for properties analysis and drying experiment. Then, the procedure for properties analysis is presented. The methods for drying experiments involved various methods used to dry the sago starch samples such as sun, oven, microwave, and freeze dry. Then the drying experiments focused on oven drying with various variable changes as well as their parameters. Finally, the methods used to analyze the drying data is presented. The experimental design is given in Figure 3.1 below.

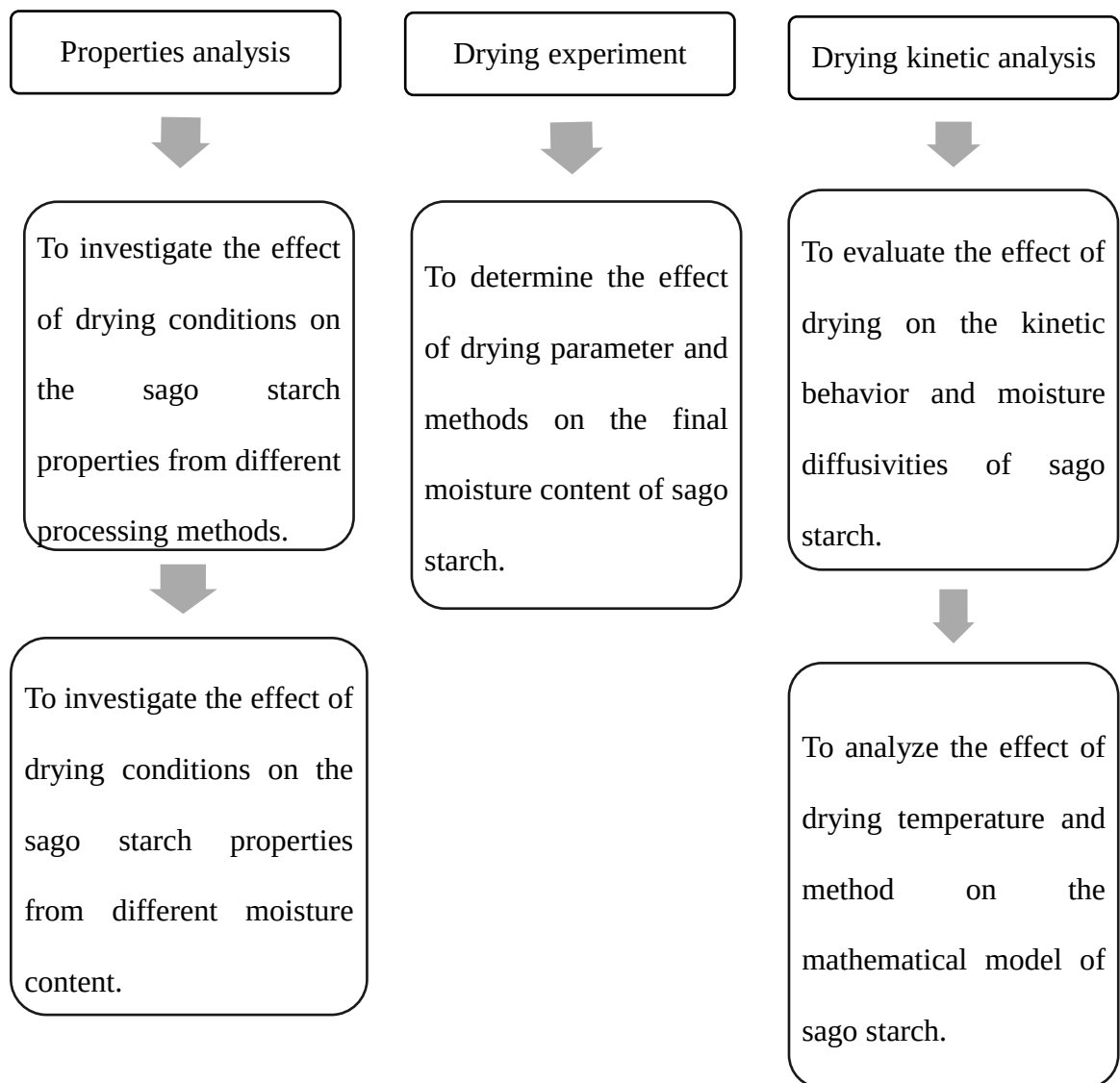


Figure 3.1: Experimental design for the study on properties and drying kinetic properties of Sarawak sago starch

3.1 Sample Preparation

3.1.1 Sago Flour

Three samples were used in this work, as shown in Figure 3.2 below. The first sample was sago flour, second is lemantak, and the third sample is fresh sago extracted in the lab. Sago flour used was bought from local supermarket in Samarahan, Sarawak (Figure 3.2a).



(a)



(b)



(c)

Figure 3.2: Samples used for starch characterization.
(a) Sago flour (b) Lemantak (c) Fresh sago starch

3.1.2 Lemantak

Meanwhile, lemantak bought was sold in clear plastic as can be seen in Figure 3.2 (b) above. This lemantak was bought from the local wet market in Samarahan but was processed from Sebuyau. Both Samarahan and Sebuyau are in Sarawak, but Sebuyau is a rural area which is located about 2 hours' drive from Kota Samarahan.

3.1.3 Fresh sago starch

Fresh sago was extracted in the lab and processed manually from sago logs received from CRAUN research. Sago logs were harvested from a nearby farm in Kota Samarahan, Sarawak. The log was kept in a refrigerator at a temperature under -20°C to avoid changes in its properties until the day it is ready for processing (Ing et al., 2000). The processing method involved chopping, blending, sieving, and settling the starch which was adopted from Darma et al. (2016).

First, the sago log was chopped with knife into small pieces of around 2 cm x 2 cm cubes. Then, the chunks were inserted into a grinder (IKA® MF10 basic) as in Figure 3.3 to produce sago sawdust.



Figure 3.3: IKA® MF10 basic to grind sago log into sawdust

The sawdust was then mixed with about equivalent amount of tap water and sieved three times. The first sieving was done by using a domestic kitchen sieve and the solution was left at room temperature for about 3 hours. The second sieving was done by using fabric sieve (napkin) and the solution was left at room temperature for about 1 hour. The third sieving was done by using the 100µm sieve and the solution was left at room temperature for 30 min. The final precipitated starch is used in the drying experiment. The sample preparation can be concluded as shown in Figure 3.4.

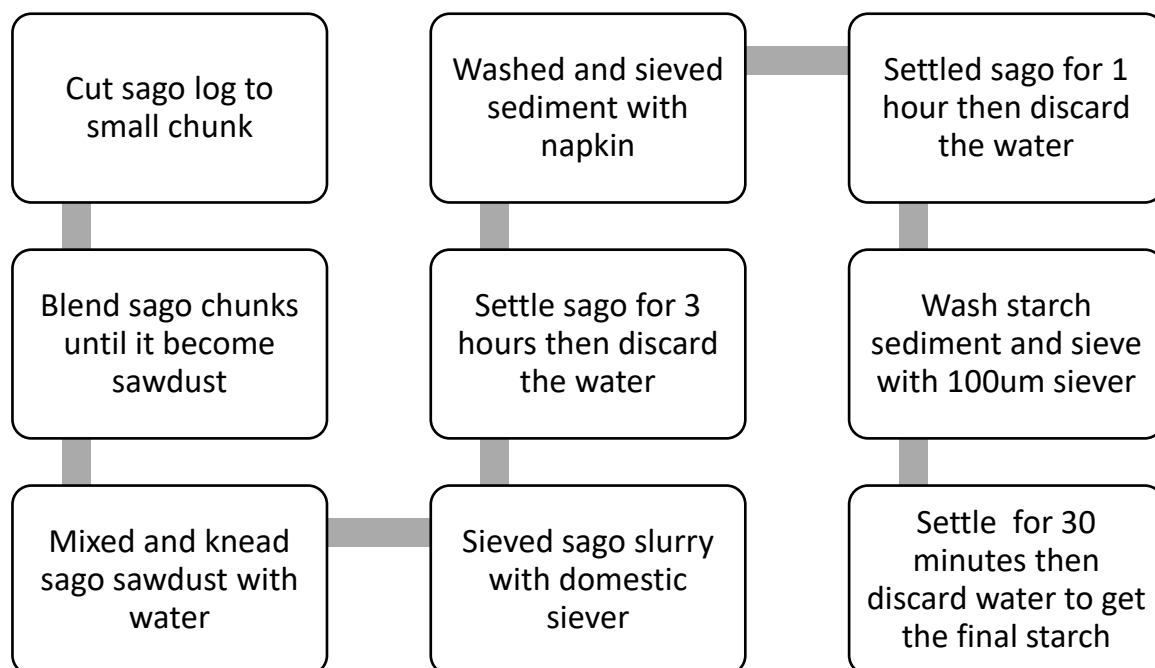


Figure 3.4: Steps for extracting sago starch

3.2 Characterization of Starch Properties

3.2.1 Chemical Properties

The chemical composition of sago starch sample was characterized through Official Methods of Analysis, 2000 (AOAC 2000).

3.2.1.1 Moisture content

The determination of moisture content in samples were done by first dry an empty dish and lid in the oven 105°C for 3 h and transfer to desiccator to cool. The empty dish and lid were weighed. Then, about 3g of sample was weighed and spread to the uniformity. The dish with sample was placed in the oven and dried at 105°C for 3 hours. After drying, the

dish was transferred with partially covered lid to the desiccator to cool. The dish and its dried sample were weighed. The moisture content was calculated as below.

$$\text{Moisture (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad \text{Equation 3.1}$$

Where W_1 is the weight (g) of sample before drying, while W_2 is the weight (g) of sample after drying.

3.2.1.2 Amylose Content

The amylose content was determined according to the method from Janaun et al. (2016). Briefly, 0.1 g of sago starch sample was added into 100 mL of volumetric flask. 1.0 mL of 95% of Ethanol and 9.0 mL of NaOH was added into the 100 mL volumetric flask. The sample was shaken well and was boiled over the shaking water bath (BS-31) for 10 minutes. After that, the solution was cooled and was made up to 100 mL using distilled water. Next, 5 mL from the 100 mL sample was pipetted out to another 100 mL of volumetric flask. Then 1 mL of 1 M acetic acid and 2 mL of I-KI was added to the solution and its volume was made up to 100 mL. The solution was shaken and stood for 20 minutes, and the absorbance of the solution was determined at a wavelength of 620 nm using UV-Vis Spectrophotometer JASCO V-650. The amylose concentration of the sample was determined from the standard curve.

3.2.1.3 Protein Content

The protein content was determined through the Kjeldahl method with 6.25 of protein-nitrogen conversion factor. About 0.5 to 1.0 gram of sample was placed in a digestion flask. 5 gram of Kjeldahl catalyst and 200 ml of concentrated H_2SO_4 were added to the sample. A tube containing the above chemical was prepared as a blank. The flask is

placed in an inclined position and heated gently until the solution becomes clear. The flask is cooled, and 60 ml distilled water is added. Immediately, the flask is connected to the digestion bulb on condenser. With the tip of condenser is immersed in standard acid, and 5 to 7 drops of mix indicator in receiver. The flask is rotated to mix the content thoroughly. It is heated until all NH_3 is distilled. The receiver is moved, the tip of condenser and titrate excess standard acid distilled is washed with NaOH solution. The calculation of protein content is measured below.

$$\text{Protein (\%)} = \frac{(A - B) \times N \times 1.4007 \times 6.25}{W} \quad \text{Equation 3.2}$$

Where, A = volume (ml) of 0.2 NHCl used sample titration

B = volume (ml) of 0.2 NHCl used blank titration

N = Normality of HCl

W = weight (g) of sample

14.007 = atomic weight of Nitrogen

6.25 = the protein-nitrogen conversation factor for fish and its by-products

3.2.1.4 Lipid content

About 20 gram of sample was homogenized with 16 mL of distilled water, 40 mL of chloroform, and 80 ml of methanol at the speed of 9500 rpm for 1 min at 4 °C. After that, 40 ml of chloroform was added and homogenized for 30 sec. Then, 40 mL of distilled water was added and homogenized again for 30 sec. After centrifugation of the homogenate at 2000 rpm at 4 °C for 20 minute, the supernatant was transferred into a separator funnel and

allowed to separate. The lipid content was determined gravimetrically by measuring triplicate aliquots of the chloroform layer into tared containers. The solvent was evaporated and weighed. The lipid content was calculated.

3.2.1.5 Ash Content

The crucible and its lid were placed in the furnace at 550°C overnight to ensure that impurities on the surface of crucible are burned off. The crucible was cooled in the desiccator for 30 minute. The crucible and its lid were weighed at 3 decimal places. About 5 gram of sample was weighed in the crucible. The sample was heated over low Bunsen flame with the lid half covered. When fumes are no longer produced, the crucible and its lid are placed in furnace. It was heated overnight without covering it with a lid. The lid was placed only after the heating was completed to prevent loss of fluffy ash. It was then cooled down in the desiccator. The ash was weighed with crucible and lid when the sample turns gray. If the sample is not gray, it should be returned together with the crucible and lid to the furnace for further heating. The ash was calculated as below.

$$\text{Ash (\%)} = \frac{\text{Weight of ash}}{\text{Weight of sample}} \times 100 \quad \text{Equation 3.3}$$

3.2.2 Resistant Starch Content

The experiment was done according to AOAC 2002.02 and AACC 38-40.01 as referred by Arshad et al. (2018). Total starch assay procedure was done with starch assay kit (Megazyme Amylo-glucosidase / alpha-Amylase Method. First, the samples were digested as the non-resistant portion of the starch were solubilized and hydrolyzed to become D-glucose. The digestion steps included sample`s incubation in a shaking water bath with pancreatic α -amylase and amylo-glucosidase (AMG) for 16 hours at room

temperature. An equal volume of ethanol was added to terminate the reaction. The resulted RS was centrifuged and recovered as a pellet. The RS pellet was then dissolved in 2M potassium hydroxide by vigorous stirring in an ice water bath over a magnetic stirrer. The solution was then neutralized with acetate buffer and the starch was quantitatively hydrolyzed to glucose with AMG. The RS content of the sample was measured as D-glucose with glucose oxidase/peroxidase reagent.

3.2.3 Granules Morphology

Granule's morphology was examined using a Scanning Electron Microscope (SEM) TM3030, HTACHI, Japan. This procedure was done by the help of a trained technician. A thin layer of starch granule was mounted on a specimen holder and the specimen holder was loaded in a sputter coater. Once metalized, the sample was viewed under 500x and 2000x magnification. All optical measurements were performed at room temperature under ambient conditions (He et al., 2020).

3.2.4 Particle Size Distribution

The mean size distributions of samples were measured by using CILAS 1090 Laser Particle Size Analyser, Orleans, France, at room temperature. Deionized water was used as the medium for wet size measurement of flours. Sonic measurements were performed to avoid the aggregation of samples (He et al., 2020).

3.2.5 Color

Color analysis of the sago samples was carried out by taking their image by using Canon EOS-1D X Mark II DSLR camera and was analyzed by using Mat-lab software

R2012b; that gives the pixel data of samples image in RGB value. The flow chart of color analysis is given in Figure 3.5 below.

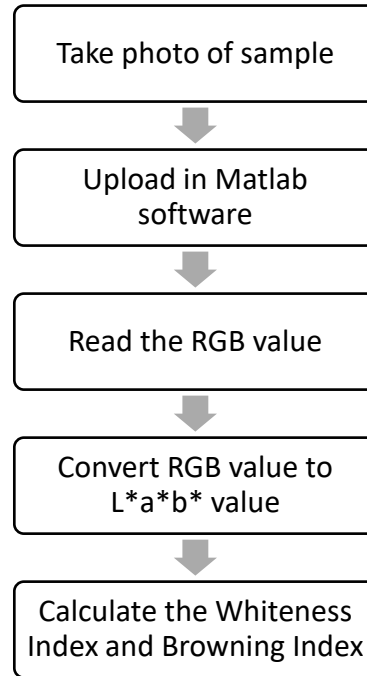


Figure 3.5: Steps for measuring sago starch color (Koleda, 2019)

Before reading the RGB value of the sample, a reference standard is decided by using white A4 paper with 100 is referred to white. 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 for ‘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 was calculated according to Yadav et al., (2006), while Browning index (BI) was calculated from Diamante et al. (2010), as in Equation 3.4 and 3.5, respectively.

$$WI = 100 - \sqrt{(100 - L)^2 + a^2 + b^2} \quad \text{Equation 3.4}$$

$$BI = \frac{[100(x - 0.31)]}{0.17} \quad \text{Equation 3.5}$$

$$\text{Where } x = \frac{(a+1.75L)}{5.645L+a-3.012b} \quad \text{Equation 3.6}$$

3.2.6 Peak Viscosity

The peak viscosity analysis was performed based on method from MS470 (1992). The starch samples were examined in a Brabender Amylograph (Brabender GmbH, Duisburg, Germany) and pasted at a heating rate of 1.5°C/min from 35 to 95°C, held 15 minutes at the 95°C and cool down to 50°C. The initial gelatinization temperature, peak viscosity, breakdown viscosity and setback viscosity were determined from the amylograph curve (Brasoveanu et al., 2013).

3.2.7 Molecular Characterization

The molecular absorption of sago starch samples was recorded on a Shimadzu Fourier Transform Infrared (FTIR) IRAFFINITY-1, Kyoto, Japan (Appendix). 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 that is around the mid-infrared region. The ATR mode was utilized along the measurement. Spectra obtained were transferred into a data analysis package. All optical measurements were performed at room temperature under ambient conditions (Hartningsih et al., 2020).

3.2.8 Thermal Stability

Thermogravimetric analysis was conducted with SHIMADZU DTG-60 H, Kyoto, Japan. About 5 mg of sample was used for each measurement. Sample decomposition was performed from 25°C to 600°C at a heating rate of 10°C/min under nitrogen atmosphere. The continuous weight loss and temperature were recorded and analyzed (Liu et al., 2013).

3.2.9 Statistical Analysis of the Results

All analysis were carried out in triplicate ($n=3$) and data expressed as means $\pm$ standard deviation (SD). One-way analysis of variance (ANOVA) single factor was performed to calculate significant differences in multiple treatment group's means through Microsoft Excel. A significant level of $p \leq 0.05$ was applied. Furthermore, to determine the differences between each group with another groups, a post-hoc t-test was applied to the data. The significance level is divided by the number of variables or groups. One group is considered significant with the other group if the p-value is less than the significance level (0.05). The details results are presented in the Appendices (Zong et al., 2020).

3.3 Drying Experiment

During all drying experiments, the initial weight (W_i), weight at interval time (W_t) and weight (W_e) at equilibrium weight was recorded. The equilibrium weight was recorded when the experiment is finished, that is when the sample weight is similar in three continuous times. The dried sample was transferred to oven drying at 105°C for 24 hours to determine its final weight (W_f). All experiments were performed in three replications. The moisture content was calculated on a dry basis as shown below (Caparanga et al., 2017).

$$M_i = \frac{W_i - W_f}{W_f} \quad \text{Equation 3.7}$$

$$M_t = \frac{W_t - W_{t-(t-1)}}{W_f} \quad \text{Equation 3.8}$$

$$M_e = \frac{W_e - W_f}{W_f} \quad \text{Equation 3.9}$$

Where M_i is the initial moisture content, M_t is the moisture content at t time, M_e is moisture content at equilibrium, while M_f is the moisture content of sample after dried at 105°C. W_i is the weight (g) of sample before drying, W_t is the sample weight (g) at t time, W_e is sample weight (g) at equilibrium, while W_f is the weight (g) of sample after dried at 105°C.

3.3.1 Oven Drying

A forced convection oven from BINDER was used to perform drying experiments with various variables and parameters (Caparanga et al., 2017). The controlled variables are time, temperature, air velocity, sample location and sample thickness. Briefly, about 50g of sago starch was filled in an aluminum foil-container and put in a preheated oven at position C in Figure 3.6.

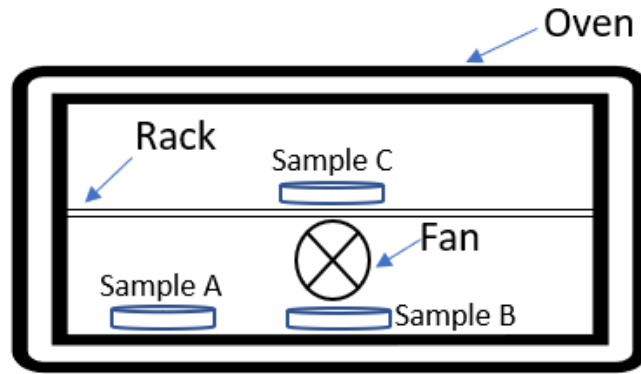


Figure 3.6: Location of sample inside drying oven

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 samples are ready for subjecting into properties analysis. The moisture content was then confirmed by using Equation 3.5 to 3.7. The dried sago starch was weighed every hour until desired moisture content was reached or until it reaches equilibrium moisture content.

As for time variable, three samples were dried in 40 °C with constant fan speed of 0% and sample location at C. The dried sago starch was weighed every hour until 5, 10 and 15 hours of drying, respectively.

For temperature variable, the drying experiments were conducted at 50, 60, 70 and 80°C (Wang et al., 2021). The air velocity, sample location and sample thickness were constant. The sago starch was weighed every hour until a constant mass was reached. The equilibrium moisture content (M_e) of the sago starch at various drying temperature was determined.

For velocity parameter, a forced draft oven was used. Fan is an important parts for air ventilation in an oven. Unlike convection oven illustrated in Figure 3.10 above, two fans used in this experiment are located at the bottom and at the top of oven for air entrance and air escape purpose, respectively. According to Bradley (2010), the air movement inside a forced oven is horizontal across shelving. The drying temperature was fixed to 60°C and the oven fan speed was controlled from 0 to 100%. In this experiment, the fan speed was set to 0, 40, 70 and 100%. The calculation of air velocity is attached in the Appendix. The air velocity was calculated as 0, 0.386, 0.676 and 0.965 m/s respectively. The sago starch samples were weighed every hour until a constant mass was reached. The equilibrium moisture content (M_e) of the sago starch at various drying air velocity was then determined (Caparanga et al., 2017).

For thickness variable, the samples varied from $\pm 30\text{g}$, $\pm 80\text{g}$ and $\pm 150\text{g}$, for thickness of 0.5cm, 1 cm and 2 cm, respectively. The samples were placed at location C with constant drying temperature of 60 °C and the air fan speed was set at 40% (0.386m/s). The sago starch samples were weighed every hour until a constant mass was reached. The equilibrium moisture content (M_e) of the sago starch at various sample thickness was determined (Kumar & Tiwari, 2007; Boutelba et al., 2018).

Generally, all samples were placed at the location same with Sample C as shown in Figure 3.6. However, for sample position variable, three sago starch samples were used and located the position same with Sample A, B and C as shown in Figure 3.6 above. Sample C is located at the middle-top of the oven, while sample A and B are located at low-side of left and right, respectively. All samples have similar weight of $\pm 50\text{g}$ and were dried at constant temperature (60 °C) and constant air fan speed at 40% (0.386m/s). The sago starch samples were weighed every hour until a constant mass was reached. The equilibrium moisture

content (M_e) of the sago starch at various sample positions was then determined (Lamlert, 2010).

For intermittent drying, about ± 50 g of sago starch samples were dried at 60 °C with 40% fan speed at location C in the oven. The on/off time was set at every one hour and the weight was also recorded every hour until it reached constant weight. The equilibrium moisture content (M_e) of the sago starch was determined (Baini & Langrish, 2006).

3.3.2 Sun Drying

For sun drying experiment, about ± 50 g sago starch was filled into an aluminum foil-container and put under the sun from eight o'clock in the morning until six o'clock in the evening every day until it reaches equilibrium moisture content. The moisture content was determined by weighing the sample weight every hour and calculated by using Equation 3.1. The samples thickness was 0.5cm. The temperature outside ranged from 34°C to 39°C with humidity recorded as 36.6%. The equilibrium moisture content (M_e) of the sago starch was determined (Arslan & Ozcan, 2010).

3.3.3 Microwave Drying

A steam convection microwave oven from Panasonic (Model NN-CS 5995) was used for microwave drying experiment. About ± 50 g sago starch was filled into a plastic container and dried inside the microwave until it reaches equilibrium moisture content. The moisture content was determined by weighing the sample weight every two minute and calculated by using Equation 3.1. The samples thickness was 0.5cm. The sago starches were dried at the lowest power of 200W to prevent caramelization (Brasoveanu & Nemtanu, 2013). The equilibrium moisture content (M_e) of the sago starch was determined (Surendhar 2018).

3.3.4 Freeze Drying

For freeze drying experiment, a freeze-dryer (Freezone[®] Plus TM 4.5 Liter cascade Freeze Dry Systems 7382033) from Labconco, USA was used. About ± 50 g sago starch was filled into a vacuum-glass tube and dried inside the freeze-dryer until it reaches equilibrium moisture content. The samples were placed in a tube at temperature of -92°C for one hour before the drying experiment as shown in Figure 3.7 (Khoozani et al., 2019).

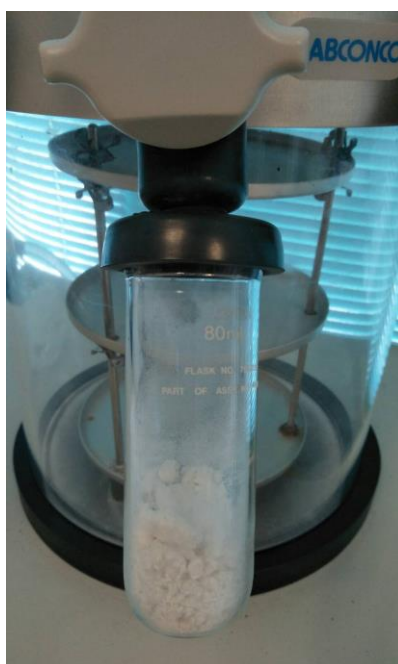


Figure 3.7: Sago sample in a tube

The moisture in the tubes was frozen to form solid ice during this period. Drying was performed at a temperature of 40°C and continued over a period of eleven hours (Khoozani et al., 2019).

3.4 Drying Kinetic Analysis

In this section, several thin drying models as listed in Table 2.1, (Chapter 2, section 2.6.1, page 41), simplified Fickian model and characteristic drying curve model were used. For each model, the fitted parameter for temperature were 50, 60, 70 and 80°C. Regression analysis was performed using Microsoft excel and the drying constant of each drying model are determined (Caparanga et al., 2017). The most appropriate model for drying sago starch was chosen based on the lowest standard error and the highest coefficient of determination (R^2). The highest coefficient of determination (R^2) is calculated by using correlation formula (=CORREL) in Microsoft Excel. Standard error (SE) which means the deviation between predicted and experimental values was then calculated as in Equation 3.10 below to confirm the model.

$$\text{Standard error, (SE)} = \left[\frac{\sum_{n=1}^i [(X_{Exp} - X_{Pred})^2]}{n-1} \right]^{\frac{1}{2}} \quad \text{Equation 3.10}$$

(Baini & Langrish, 2007)

Upon determination of moisture diffusivities, calculation of activation energy is necessary since it could tell the sensitivity of the diffusivity against temperature in positive correlation (Maskan et al., 2002). Activation energy, E_a (kJ/mol) for moisture diffusivity is calculated by using Arrhenius-type equation below.

$$D_{eff} = D_0 \exp \left[\frac{-E_a}{R_g T} \right] \quad \text{Equation 3.11}$$

$$(\ln(D_{eff}) = \ln(D_0) \exp \frac{-E_a}{R_g} \left[\frac{1}{T} \right]) \quad \text{Equation 3.12}$$

Where D_0 is the pre-exponential factor of Arrhenius equation (m^2/s), R_g = is the constant of perfect gases (8.3143 kJ/kmol.K) and T is the drying temperature in Calvin. Furthermore, Equation 3.11 can be rearranged as in Equation 3.12 to determine E_a through the slope of the Arrhenius curve representing $\ln(D_{\text{eff}})$ against $1/T$ (shown in Appendix).

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter presents results and discussions that are separated into six sections. The first section is about the characterization of three types of sago starch that are lemantak, sago flour, and fresh sago starch. In section one, several properties were analyzed such as the composition, morphology, particle size, color, viscosity, molecular structure and thermal stability. The second section is about the effect of heating to sago starch prepared in laboratory in which four samples with different moisture content were subjected to properties analysis like those in section one. The third, fourth and fifth section discussed on the EMC, drying kinetics and moisture diffusivities which includes the effect of different drying temperature, air velocity, sample thickness, sample position, as well as different drying methods. Finally, the assessment of drying models for sago starch is discussed. Three main drying concepts were assessed that are empirical model, Fick Diffusion model and the Characteristic Drying Curve model. For empirical model, ten thin layer drying model were applied, while for Fick Diffusion model, the simplified version was applied.

4.2 Characterization of Processed Sago Starch

In this section, chemical composition, physicochemical and functional properties analysis of three processed sago starch samples are presented. The analysis involves proximate composition, morphology, color, resistant starch content, peak viscosity, thermal stability, granule size and molecular structure.

4.2.1 Chemical Properties of Sago Starch Samples

Table 4.1 shows the proximate composition of three sago starch samples; lemantak is the sago starch prepared traditionally obtained from local market, sago flour is the commercial sago starch and fresh refers to the sago starch prepared in the lab. The composition measured were moisture content, starch, amylose, fat, protein, and ash content.

Table 4.1: Chemical composition of sago starch from different processing methods

Composition (d.b.*)	Lemantak	Sago flour	Fresh sago
Moisture Content [%]	22.44 ± 2.07a	13.27 ± 0.85b	32.11 ± 2.17c
Starch [%]	81.41 ± 1.22a	57.16 ± 2.88b	80.03 ± 0.72c
Amylose [%]	27.27 ± 1.21a	15.73 ± 0.59b	26.69 ± 2.78c
Fat [%]	0.26 ± 0.05a	0.17 ± 0.03b	0.32 ± 0.03c
Protein [%]	0.08 ± 0.01a	0.06 ± 0.01a	0.07 ± 0.01a
Ash [%]	0.23 ± 0.01a	0.19 ± 0.00a	0.28 ± 0.01b

*Reported values are the mean ± Standard Deviation, SD (n=3). The lower-case letters indicate significant differences ($p \leq 0.05$) within the same row, generated by ANOVA

4.2.1.1 Moisture Content

The moisture content of sago flour is the lowest, which is 13.27%, has slightly exceeds the limit of Malaysia Standard edible sago starch grade which is stated as 13% maximum, but comply with the non-edible grade of 15% maximum (Karim et al., 2008). The low moisture content allows sago flour to be kept for a longer period. Meanwhile, the

moisture content of lemantak and fresh sago starch are 22.44% and 32.11%, respectively. Both values are very high and exceeded the standard limit for edible and non-edible grades of sago starch. Moreover, high moisture content encourages the microbe's activities and shortens the shelf life. The fresh sago starch was not subjected to drying process, while traditional sago starch has been dried through sun drying process. The high standard deviation of lemantak moisture content indicated that sun drying process are not well monitored. In addition, Singhal et al., (2008) had addressed that the high moisture content in traditional sago starch has potential to lead to serious problem such as microbial activity spoilage and international marketing limitation.

Besides, the results of moisture content for lemantak and fresh sago starch have relatively high standard deviation that is 2.07 and 2.17, respectively. This indicates their granules' sensitivity in absorbing moisture from the air. The results show that sago starch processed traditional (lemantak) and manually (fresh sago) has more tendency to absorb moisture as compared to machine processed sago starch. Uthumporn, Wahidah and Karim (2014) also suggested that sago starch granule has different tendencies to absorb moisture from the air depending on their growth stages.

4.2.1.2 Starch Content

Table 4.1 shows lemantak has the highest starch content (81.41%), followed by fresh sago (80.03%), and sago flour (57.16%). that all amylose content of sago starch samples is not dominant to amylopectin. Even though the starch content of sago flour is relatively low than Lemantak and fresh sago starch, there is no standard for starch content of edible starch. However, the huge difference of starch content between sago flour and both lemantak and fresh sago starch has clearly indicated the effect of extreme process to the quality of sago

starch. It is suspected that machinery and chemical processes involved in extraction, purification and drying process of sago flour has contributed to this results. In terms of nutritional value, lemantak and fresh sago starch provide more carbohydrate than sago flour.

4.2.1.3 Amylose Content

Sago flour has the lowest amylose content at 15.73%, while Lemantak and fresh sago have higher, 27.27% and 26.69%, respectively. As amylose plays a major role in flavor and odor binding capacity, this result might influence the intensive flavor of favorite lemantak meal eaten by local people, at the same time can explain the moldy smells in lemantak compared to odorless sago flour (Sayaslan, 2006). In addition, according to Hasjim (2009), starch with amylose content less than 50% are considered not waxy or viscous. Moreover, the results of both starch and amylose content show the lowest value in sago flour as compared to the other starch. The results of starch content and amylose content are in similar pattern since amylose is a part of starch content.

4.2.1.4 Fat Content

Table 4.1 also shows that the fat content of lemantak, sago flour and fresh sago starch are very low, that is 0.26%, 0.17% and 0.32%, respectively. Fat content is an undesirable content in starch, thus low-fat content indicates high starch purity. There is no standard for sago starch fat content, but the values obtained in this result are within the range with the results obtained by Uthumporn et al. (2014) and Ahmad et al. (1999). Sago flour has the lowest fat content which is possibly affected by its processing method such as extraction and drying process. Soxhlet Extraction method is often used in modern starch factories to remove surface fat molecule or usually termed as lipid (Uthumporn et al., 2014).

As for the drying process, high drying temperature applied in modern factory has tendency to induce granules swelling which may lead to penetration of lipid from starch granule surface into the granule, which will results in the formation of amylose-lipid complex (Ellis et al., 1998). Amylose-lipid complex is resistant towards enzymatic reaction, thus make it prone to lipid detection which can affect the experimental results (Birt et al., 2013). Nevertheless, the presence of amylose-lipid complex is beneficial to sago starch in a way that it can improve the odor and textural properties during food manufacturing (Zaidul et al., 2003; Uthumporn et al., 2014).

4.2.1.5 Protein Content

Similar to fat, protein is also a minor component in starch. Table 4.1 shows that the protein content in lemantak, sago flour and fresh sago starch is 0.08%, 0.06% and 0.07%, respectively. However, Ahmad et al. (1999) obtained higher protein value which is around 0.13% to 0.25%; while Adawiyah et al. (2013) obtained similar protein content that is below 0.1%. These small percentage is considered very low and negligible which explains why sago starch is categorized as gluten-free starch (Mohamed et al., 2008). Gluten is a type of starch abundant protein that can affect starch elasticity. Gluten-free starch is safe and suitable for gluten intolerance people (Scherf et al., 2016). An example of starch that contains high amount of protein or gluten is wheat starch which consists of 13.2% of protein according to Zaidul (2001). Recent work from Hu et al.(2017) had successfully removed surface protein and lipid from rice starch; thus, giving hope for others gluten starch to be used more safely and widely. However, protein removal technique is still limited to surfactant molecules because the protein molecules inside the starch granule are more complex (Horstmann et al., 2017).

4.2.1.6 Ash Content

The ash content of lemantak, sago flour and fresh sago starch is 0.23%, 0.18% and 0.28%, respectively. All the results are compliant with the required Malaysia Standard of 0.5% maximum. Ahmad et al. (1999) also had obtained similar value range within 0.06% and 0.43%. High ash content in lemantak is also probably due to the water quality used in traditional processing at rural area (Kamal et al., 2007). In the same manner, low ash content in sago flour indicates high water quality used in modern factory. Besides, low ash content in sago flour can be the result of purification process from sulfur dioxide commonly used in modern factories to enhance color and remove impurities but can adversely affect starch quality. However, sulfur dioxide treated starch might not be suitable to some people that has allergy reaction towards the substance (Sindu, 2019). In addition, ash content also might be affected by the composition of soil surrounded the originated sago palm (Ahmad et al., 1999); or by mistakenly harvesting immature palm (Karim et al., 2008)

4.2.2 Resistant Starch Content of Sago Starch Samples

Resistant starch is an important quality attribute that determines the acceptance of sago starch by consumers (Birt et al., 2013). It has been proved in several literatures as beneficial to human health and effective to diabetic people (Capriles et al., 2008). This is due to its behavior that is able to escape digestion in small intestine but fermented in the intestine. The resistant starch value of traditionally processed sago starch (lemantak), commercial sago starch (sago flour) and lab-scale extracted (fresh sago starch) samples were analyzed in this study and the results are shown in Table 4.2.

Table 4.2: Resistant starch value of lemantak, sago flour and fresh sago

Sample	Resistant starch value (%)
Lemantak	62.30 ± 0.12a
Sago flour	57.20 ± 0.34b
Fresh sago	59.91 ± 0.21c

*Reported values are the mean ± Standard Deviation, SD (n=3). The lower-case letters indicate significant differences ($p \leq 0.05$) within the same row, generated by ANOVA.

Lemantak shows significantly high resistant starch content, followed by fresh sago starch and finally, sago flour. The resistant starch content in lemantak is 62.30%, followed by fresh sago that is 59.91%, and sago flour that is 57.20%. Amylose gives more roles to determine the resistant starch behavior than amylopectin due to its characteristic of tightly packed linear polymer. Therefore, the resistant starch results in Table 4.2 are positively correlated with amylose content result in Table 4.1. Resistant starch content in lemantak is higher than fresh sago probably due to the sun drying process that involved natural heating and cooling effect which may physically modify the resistant starch. However, it was found that extreme heating and cooling of retrogradation-induced process will result in inverse effect (Arshad et al., 2018). Besides, sago flour has the lowest resistant starch content. It is suspected that the chemical additive in modern manufacturing had decreased the resistant starch in sago flour.

4.2.3 Granule Morphology of Sago Starch Samples

Sago starch samples were subjected to Scanning Electron Microscope at 500 and 2000 magnification for granules morphology analysis (Villadiego et al., 2020). The results from 500 magnification are shown in Figure 4.1 to 4.3.

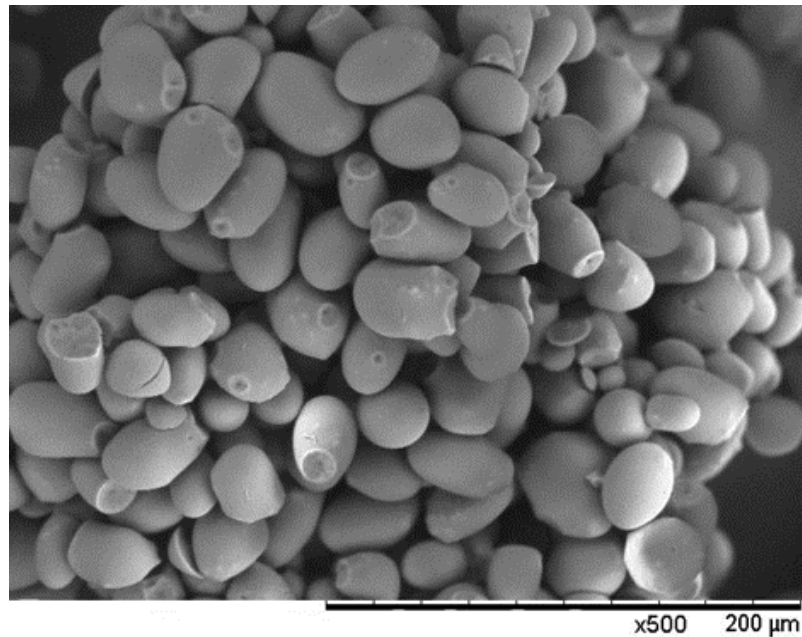


Figure 4.1: Starch granule in lemantak at 500 magnification

Lemantak granule in Figure 4.1 presents as an agglomerates which means granule clusters as the starch granules are attached to each other to become a 400μm lump. Similar results were obtained by Horstmann et al. (2017) by using tapioca and rice in which the present of starch granule's cluster has been considered as a normal phenomenon in native starch. However, the starch granule cluster can be de-attached as in ambient condition. It means that this phenomenon is different with retrogradation in gelatinization or pre-

gelatinization condition. The presence of big cluster may limit the starch acceptance for since the standard particle size shall not exceed 80 μ m, as according to Malaysia Standard.

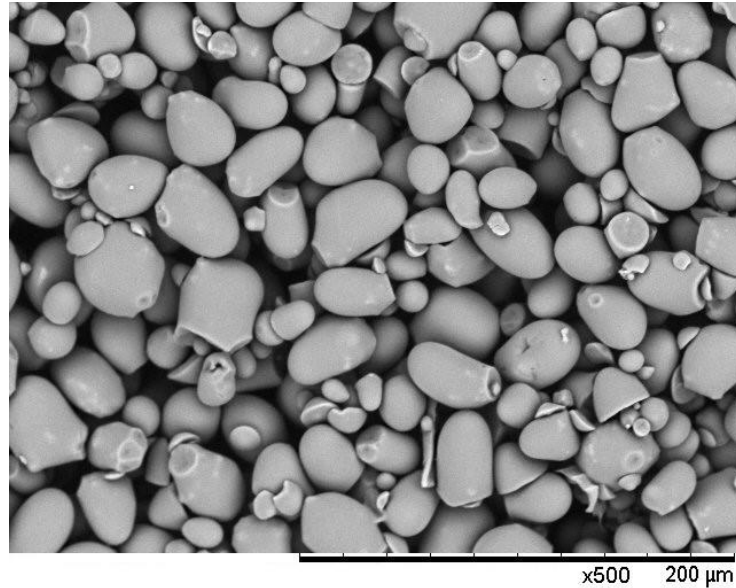


Figure 4.2: Starch granule in sago flour at 500 magnification

Meanwhile, sago flour in Figure 4.2 does not shows any starch agglomeration, but clearly shows a high number of chipped granules. Chipped granules are caused by mechanical injury probably due to the application of high-speed rasper in modern factory. The effect due to the application of high-speed rasper has been addressed by Kopli (2015) in his study. The presence of chipped granule can affect the starch digestibility by decreasing the resistant starch content (Hasjim, 2009).

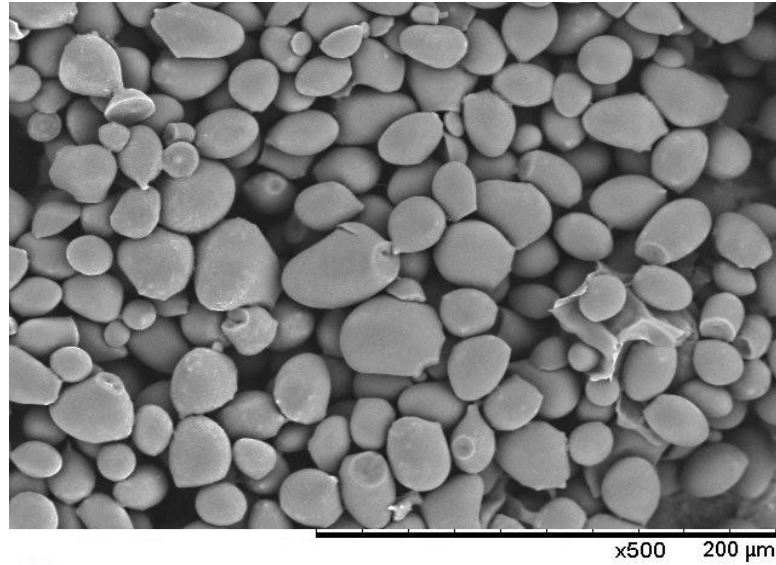


Figure 4.3: Starch granule in fresh sago at 500 magnification

Fresh sago starch in Figure 4.3 shows no agglomeration and less chipped granules but some impurities can be spotted. In modern factory, impurities are reduced by using sulfur dioxide but this process has bad effect on consumer with allergy reaction towards the substance (Jyothi et al., 2007).

Meanwhile, the results of sago starch samples morphology from 2000 magnification are shown in Figure 4.4 to Figure 4.6.

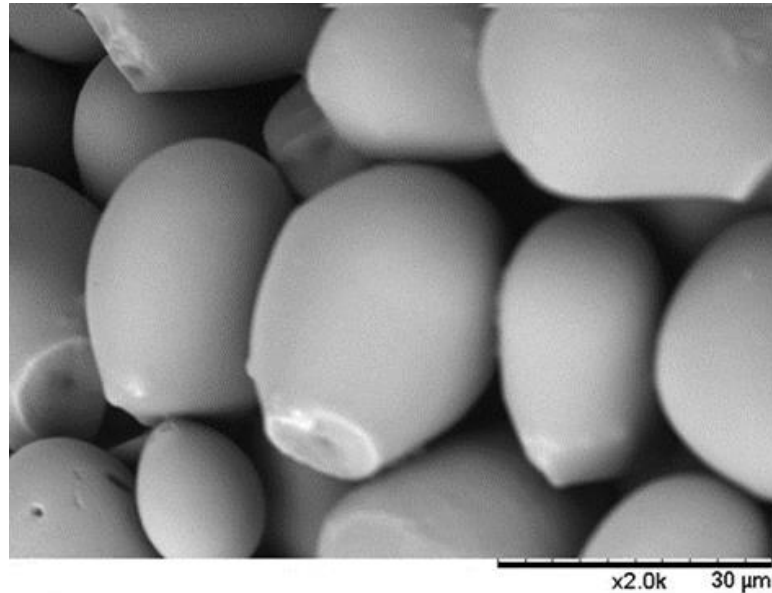


Figure 4.4: Starch granule in lemantak at 2000 magnification

Figure 4.4 shows that granules in lemantak have smoother surface when compared to granules in sago flour and fresh sago starch. This might be the effect of slow and gradual process of moisture removal during sun drying which is possibly results in smoother granules as suggested by Gouaou et al. (2016) and Tang et al. (2017).

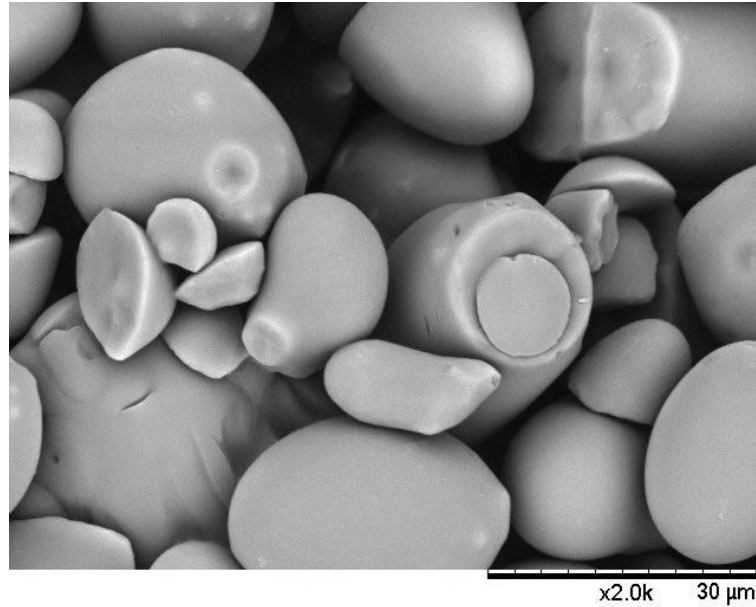


Figure 4.5: Starch granule in sago flour at 2000 magnification

As for sago flour granules in Figure 4.5, the chipped area can be seen more clearly which looks like a growth ring. Chipped granules are the results of broken or injury granule caused by harsh mechanical processes. It also can be seen that the granules are not in intact oval shape with small deformation mainly at the peripheral regions (area away from the center). This condition is as the result of partial gelatinization related to high drying temperature probably above 60°C to 90°C (Timm et al., 2020). Therefore, it is suspected that sago flour has been subjected to high temperature during machineries processing in the factory.

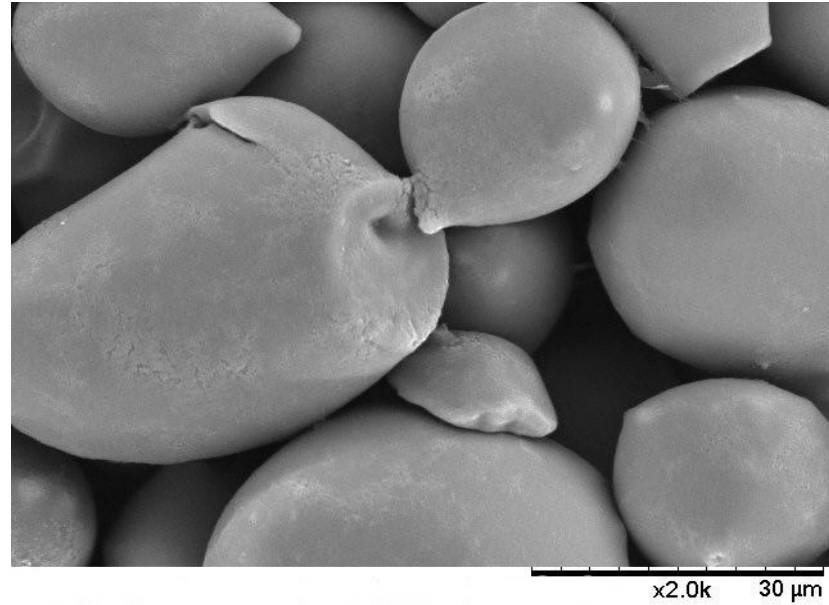
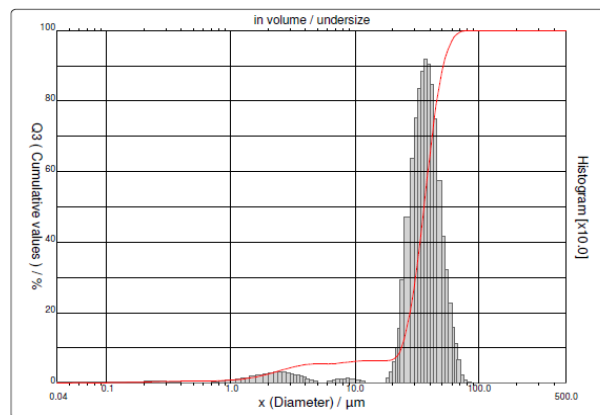


Figure 4.6: Starch granule in fresh sago at 2000 magnification

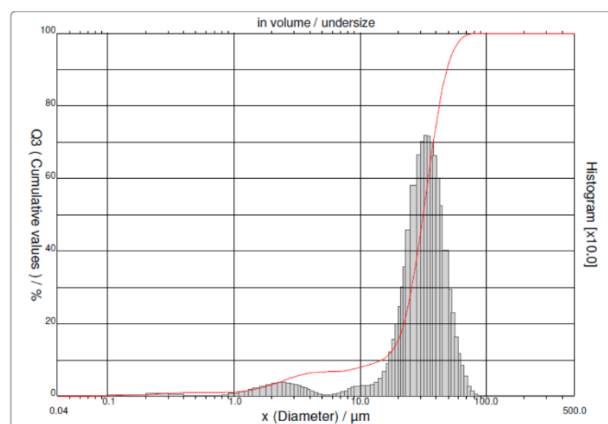
Meanwhile, magnifying image of fresh sago starch granules in Figure 4.6 shows unsmooth surface contrast with lemantak. Previous study on drying effects to morphology had indicated that drying can improve the starch granule surface to become smoother (Gouaou et al., 2016). This indicates the importance of drying process towards starch physical properties while removing extra moisture content.

4.2.4 Particle Size Distribution of Sago Starch Samples

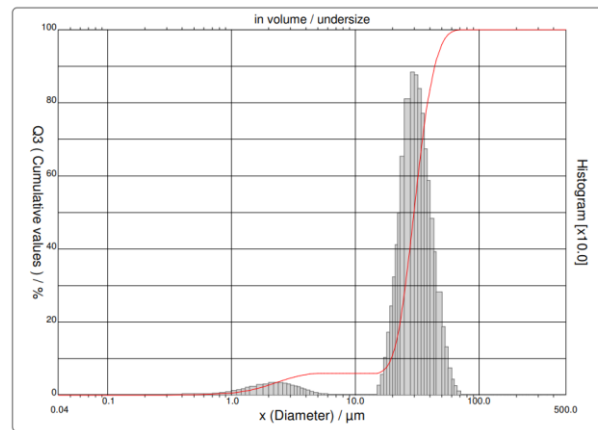
Analysis of starch granule size is important in order to determine its suitability for double modification (Wattanachant et al., 2002). Double modification of starch is implemented in order to improve any type of starch weaknesses such as low stability at high temperature, thermal decomposition and high tendency for retrogradation (Ariyantoro et al., 2018). Granule size of sago starch samples were analysed through granule size distribution data obtained from particle size analyser. The granule size distribution of lemantak, sago flour and fresh sago starch are shown in Figure 4.7 (a), (b) and (c), respectively. Generally, all samples contain of similar size that is in the range of between 1- 100 μm .



(a)



(b)



(c)

Figure 4.7: Granule size distribution of (a) lemantak, (b) sago flour and (c) fresh sago

All samples display bi-modal size distribution with the first mode between 1 to 10 μm , and the second mode between 20 to 100 μm . Generally, sago starch granule shape is known as oval shape, with average diameter of 50 μm (Ahmad, 1999). Therefore, the result indicates that sago starch granules are divided into two distributions of big and small size. The small size can be referred to chipped granules while the big size can be referred to intact granules or granule clusters. Lemantak has the highest peak distribution for big granule size which is consistent with the granule morphology result that shows lemantak has big granule cluster. Moreover, the result also shows that sago flour has the most distributions for small granule size. This is also consistent with the granule morphology result that shows sago flour has more chipped granules.

The results obtained for granule mean diameter shows that the mean diameter of lemantak is the highest, followed by sago flour and fresh sago that are 35.91 μm , 31.74 μm , and 30.19 μm , respectively. This indicates that traditional processing caused less granules damaged as compared to modern factory processing. The presence of wind or air velocity in sun drying of lemantak may also influenced the high average granule size, as per suggested

by A.R. Caparanga (2017). Meanwhile, the small mean diameter in fresh starch indicates that dried starch has bigger granule size as compared to un-dried starch. Furthermore, granule size also increases at higher heating rate as found by Patel and Seetharaman (2006).

4.2.5 Color Analysis of Sago Starch Samples

The color properties of lemantak, sago flour and fresh sago starch were investigated through color hunter and the results are shown in Table 4.3 below.

Table 4.3: Color Properties of lemantak, sago flour and fresh sago starch

Sample	L value	a value	b value	Whiteness Index
Lemantak	93.50 ± 0.42b	-0.11 ± 0.10a	3.49 ± 0.41c	92.62 ± 0.53b
Sago flour	94.44 ± 0.35c	-0.49 ± 0.44a	-0.05 ± 0.06a	94.41 ± 0.37c
Fresh sago starch	87.48 ± 0.16a	-0.27 ± 0.16a	2.53 ± 0.24b	87.17 ± 0.24a

*Reported values are the mean ± Standard Deviation, SD (n=3). The lower-case letters indicate significant differences ($p \leq 0.05$) within the same row, generated by ANOVA

L* value indicates lightness, (+ve) a* value indicates red color, (-ve) a* indicates green color, (+ve) b* value indicates yellow color, and (-ve) b* value indicates blue color. Sago flour has the highest 'L' value that is 94.44. This was followed by Lemantak and fresh sago that is 93.50 and 87.48, respectively. In addition, it was mentioned previously that the minimum requirement for L value in Malaysia Standard for edible sago starch is 90. The results for whiteness index is in similar trend with L value as both have positive correlation as shown in Equation 3.4 The whiteness index is highest in sago flour, followed by lemantak, and fresh sago as 94.41, 92.62 and 87.17, respectively. High whiteness index adds more

commercial value in starch as it indicates the high quality of starch in term of color (Hiroyuki, 2014). For instance, in Japan, white sago is sells at relatively higher price, when compared to brown sago (Singhal et al., 2008). However, Karim et al. (2008) mentioned that sago flour often faces color variety through batch to batch per production, that will affect their acceptance in some industrial applications. In order to reach the specified standard, some factory practice bleaching technique such as Sulphur dioxide, bio-bleaching and ozone treatment in order to produce whiter sago starch (Karim et al., 2008). However, the usage of these additives can cause side effects to consumers as stated previously.

Color results for lemantak show the highest 'a' and 'b' value of -0.11 and 3.49, respectively. This is followed by fresh sago starch with its 'a' and 'b' value as -0.27 and 2.53, respectively. Meanwhile, sago flour as the whitest flour has the lowest 'a' and 'b' value of -0.49 and -0.05. In visual, lemantak usually are pinkish in color, while fresh sago starch has more creamy color. The high value of 'a' and 'b' in both lemantak and fresh sago can be related to color changes and development phenomenon in sago starch known as browning. Browning is caused by immature pith, duration of extraction process, extreme drying heat and/or polyphenol oxidation (Konuma et al., 2013; Karim et al., 2008). The amount of polyphenol compound depends on soil condition in which neutral soil that contains low sulfur produce sago starch with low polyphenol content; while acidic soil from swampy area contains high sulfur that produce sago starch with high polyphenol content (Hiroyuki, 2014). Consequently, the existence of polyphenol compound cause browning in starch due to oxidation which is more likely triggered by heavy metal ions found in tap water during extraction process (Purwani & Thahir, 2006).

4.2.6 Peak Viscosity of Sago Starch Samples

In this study, peak viscosity is used to observe the viscosity of sago starch samples. Peak viscosity occurs when most granules swollen to their rigidity extent upon shear stress. The peak viscosity of lemantak, sago flour and fresh sago starch were studied through Brabender Amylogram and the results are shown in Table 4.4 below.

Table 4.4: Peak viscosity of lemantak, sago flour and fresh sago starch

Sample	Peak viscosity in amylograph unit (AU)
Lemantak	631 ± 0.19
Sago flour	584 ± 0.13
Fresh sago starch	667 ± 0.22

*Reported values are the mean ± Standard Deviation, SD (n=3). The lower-case letters indicate significant differences ($p \leq 0.05$) within the same row, generated by ANOVA

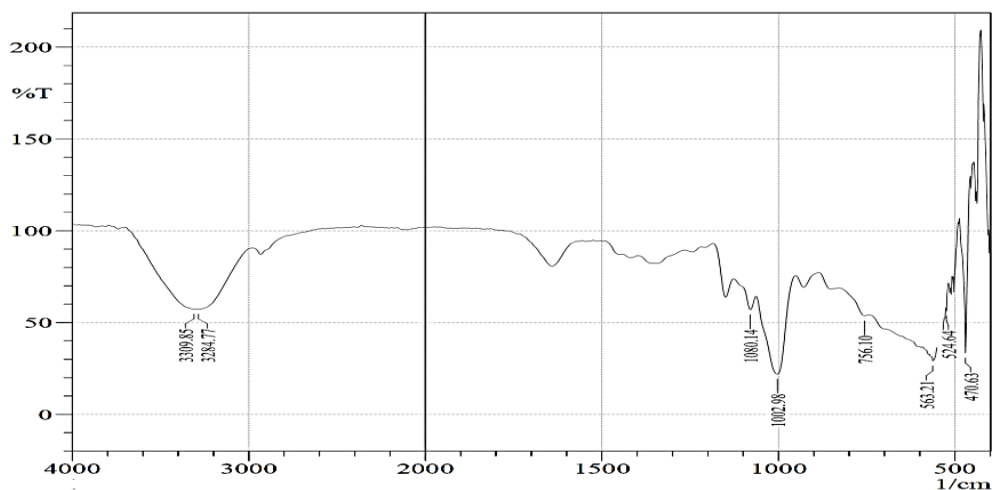
Fresh sago starch has the highest peak viscosity, followed by lemantak and finally the sago flour that significantly has the lowest peak viscosity. Starch viscosity is mostly affected by granule rigidity, but also affected by other factors such as proximate content. This result is in contrast with the results of fat content in Table 4.1 in lab sago starch that has the highest lipid content predictably will show the lowest peak viscosity since lipid can inhibit granules swelling as addressed by Alcázar-alay et al. (2015). Besides, the results in Table 4.4 also shows that undried fresh sago starch has higher viscosity than the dried sago starch. Sumadiwangsa (1995) also had found that natural sago starch has higher viscosity

than some of the chemically modified sago starch. However, most native starch show inconsistent peak viscosity probably due to the effects from its palm growth condition, which limits their industrial demands (Ellis et al., 1998 ; Alcázar-alay et al., 2015).

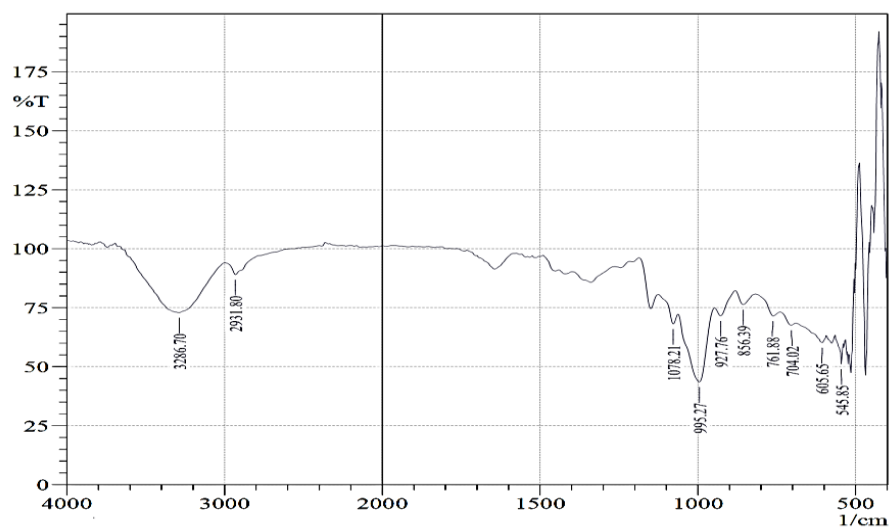
Meanwhile, sago flour shows the lowest peak, which suggests that it has degraded starch granules. Sago flour has more chipped and broken granules as compared with lemantak and fresh sago starch, which has been shown in Figure 4.2 and 4.5. This condition had caused sago flour to has low rigidity and low swelling capacity against heat and shear in excess (Othman et al., 2015). Nevertheless, the reduction in peak viscosity is demanded in weaning and supplementary foods (Yadav et al., 2006).

4.2.7 Molecular Characterization of Sago Starch Samples

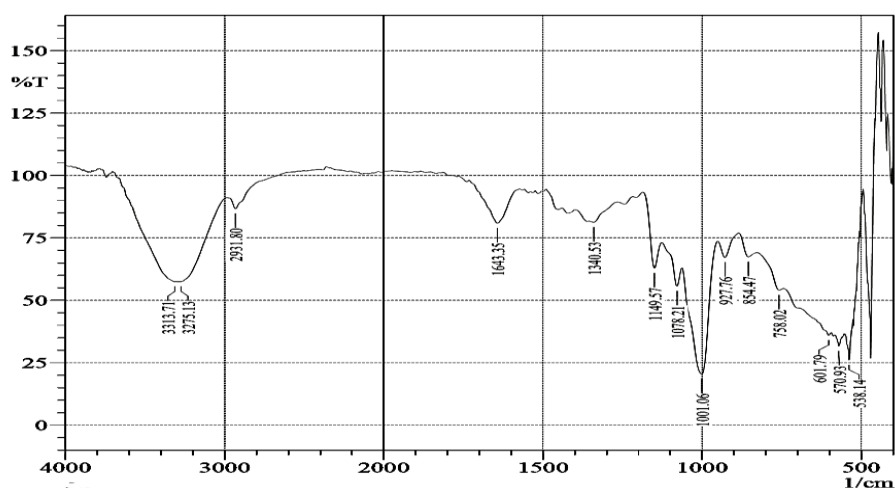
Information about sago starch molecules composition can be obtained through FTIR data, as shown in Figure 4.8 below.



(a) Lemantak



(b) Sago flour



(c) Fresh sago

Figure 4.8: Infrared spectroscopy of (a) lemantak, (b) sago flour, and (c) fresh sago

Generally, all samples show similar spectra pattern which indicate the similarity in basic molecular components. There are four major peaks observed from all spectra. There is the peak at 500 to 800 cm^{-1} , 1000 cm^{-1} , 1600 cm^{-1} , 2900 cm^{-1} and 3000 to 3600 cm^{-1} . The peaks at 500 to 800 cm^{-1} show skeletal mode vibration of pyranose ring, the peak at 1000 cm^{-1} is related to the glycosidic bond, the peak at 1600 cm^{-1} is related to the H bond stretch in starch

molecules, the peak at 2900 cm^{-1} is related to C-H bond in glucose subunit, while the peak at 3000 to 3600 cm^{-1} is related to O-H bond starch in starch (Kizil et al., 2002). The spectra similarities in skeletal and major peaks are caused by the similarities in chemical bond absorption which also indicated that all samples share similar molecule composition.

However, it also can be seen that some of the peak intensities in lemantak, sago flour and fresh sago are different. Fresh sago consistently shows the highest peak intensity at 1600 cm^{-1} and 3300 cm^{-1} , followed by lemantak, and sago flour. According to Amir et al. (2013), the peak at 1600 cm^{-1} and 3300 cm^{-1} represents the presence of water on the basis of functional group OH and H, respectively. Therefore, the results have clearly indicated that the amount of moisture content in fresh sago is the highest, followed by lemantak, and sago flour. This result is consistent with the results of moisture content in Table 4.1. In addition, Amir et al. (2013) also had successfully used FTIR to observe moisture content different in wheat varieties.

4.2.8 Thermal Stability Analysis of Sago Starch Samples

Thermogravimetric (TGA) analysis was performed to understand the thermal degradation characteristic of lemantak, sago flour and fresh sago starch. The TGA thermogram and char yields are shown in Figure 4.9.

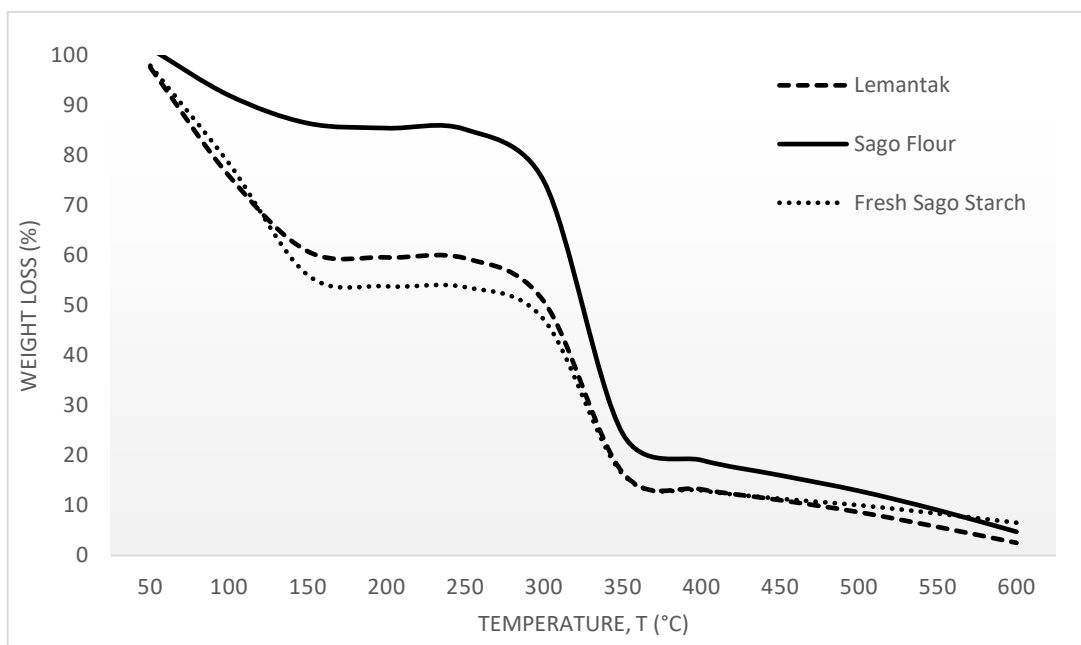


Figure 4.9: Weight loss for lemantak, sago flour and fresh sago starch

The thermogram for all samples behaved in the same manner without shifting, which indicated their similarity in terms of chemical compounds. In general, weight loss of starch can be divided into two steps. The first one is related to the evaporation of water molecules, and the second step is related to thermal decomposition.

Evaporation of water occurs at the initial stage of heating which is around 60°C and more pronounced in samples with higher moisture content that is fresh sago starch and lemantak (Piyada et al., 2013). The second phase of 110°C to 190°C is referred to the loss of bound water content and the loss of volatile compounds. The bound water content or volatile compounds can either complexed with amylose or adsorbed through granule surface to the extent of amorphous area (Sayaslan, 2006; Tian et al., 2011). In addition to this second phase, the weight loss of lemantak and fresh sago starch were more pronounced when

compared to sago flour, that indicated better thermal stability in sago flour in term of temperature range between 60°C to 190°C, which is desirable for industrial application.

The onset of 300°C caused major decomposition with maximum rate occurring at 400°C which corresponds to more complex process including dehydration of saccharide rings and de-polymerization. Both lemantak and fresh sago starch show up to 90% of weight reduction, while sago flour shows only 80% of weight reduction. Again, sago flour had showed higher thermal stability in higher temperature ranged between 300°C to 600°C. Nevertheless, fresh sago starch gave the highest char yield among all samples, which indicates that it has the strongest molecular structures, perhaps due to native crystalline characteristics (Piyada et al., 2013).

4.2.9 Concluding Remarks

The results obtained in this section show that sago starch properties are affected by the processing method. Therefore, it was clear that manual processing results in different properties of sago starch as compared with machineries processing.

4.3 Effect of Drying on Sago Starch Properties

4.3.1 Effect of Drying on Sago Starch Moisture Content

Drying reduces moisture content of sago starch as time increases, as shown in Figure 4.10. It takes about six hours to reduce the moisture content from 40% to 30%, four hours from 30% to 20%, and five hours from 20% to 10%. This result shows that it takes about an average of 5 hours for 10% reduction of the moisture content; the amount of sample used was about 50 gram. Njintang and Mbofung (2003) had also shown quite similar result for Taro flour. They estimated that the drying time required to achieve moisture content of 30%,

20% and 10% were 6, 9 and 15 hours, respectively. Findings here may indicate that sago and taro starch may have quite similar drying behavior. Here, each sample was named in regards with their moisture content which is MC40 (42.37%), MC30 (30.28%), MC20 (20.39%) and MC10 (10.90%), analysis on the effect of moisture content reduction to the physical properties of all samples is discussed in the following text.

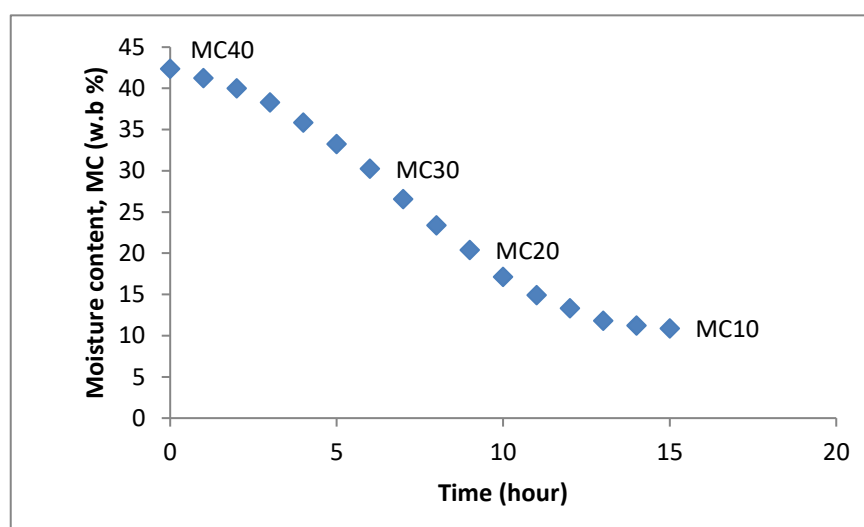


Figure 4.10: Drying profile of fresh sago starch dried in oven at 40°C

4.3.2 Effect of Drying on Sago Starch Morphology

Figures 4.11 to 4.14 show the micrograph of sago starch granules of sample MC40, MC30, MC20 and MC10 at 500 and 2000 magnification.

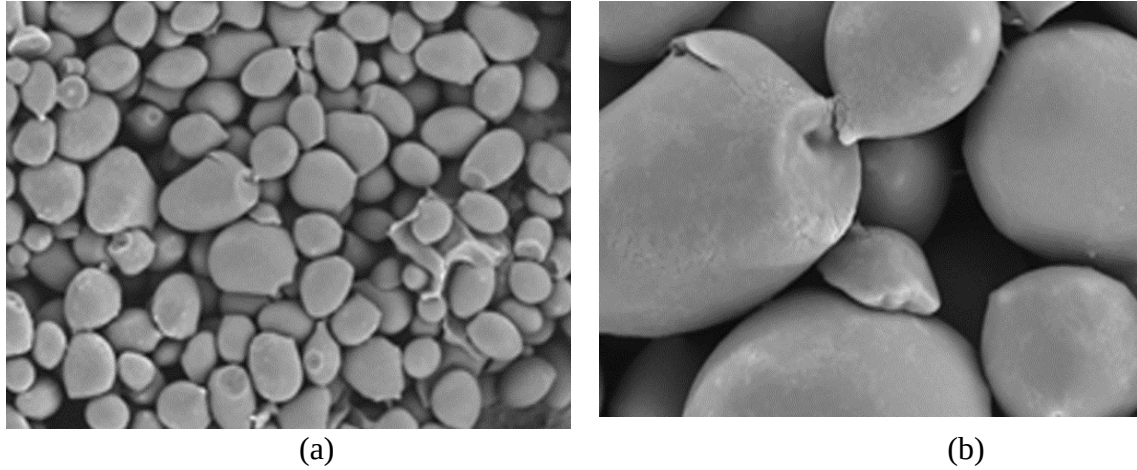


Figure 4.11: MC40 granules at (a) 500x magnification and (b) 2000x magnification

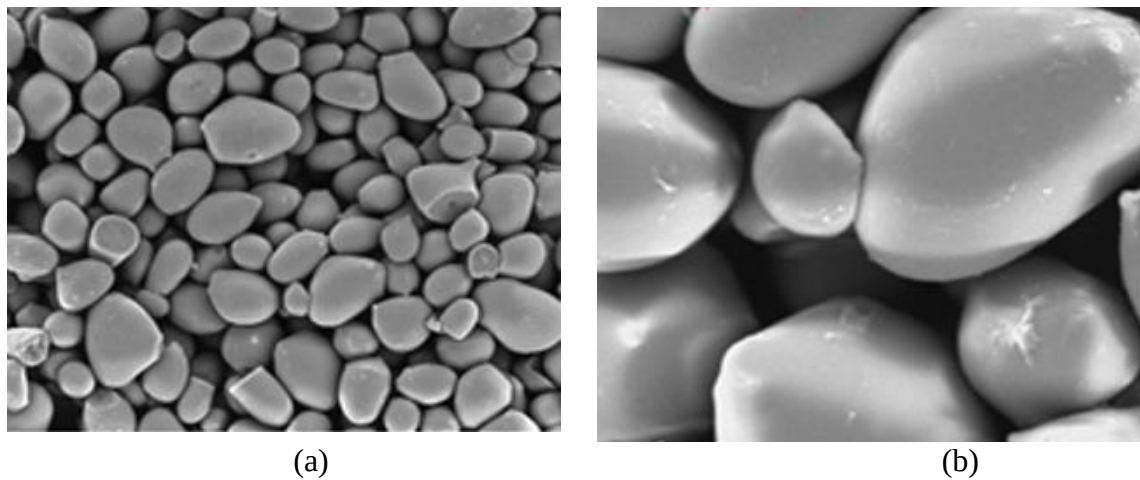
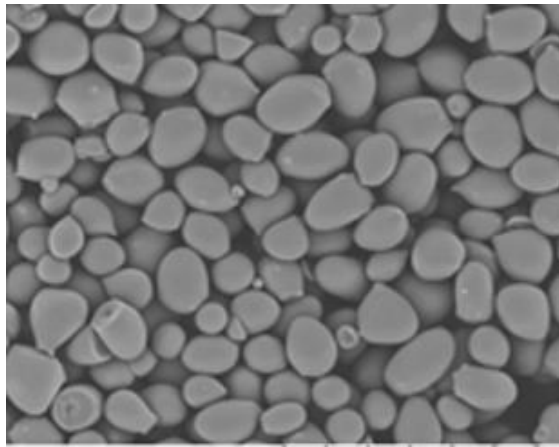
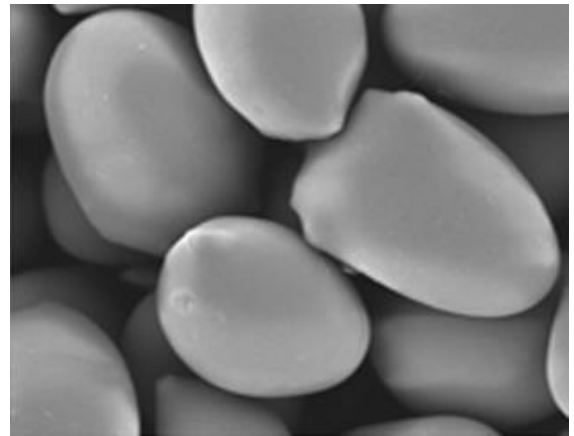


Figure 4.12: MC30 granules at (a) 500x magnification and (b) 2000x magnification

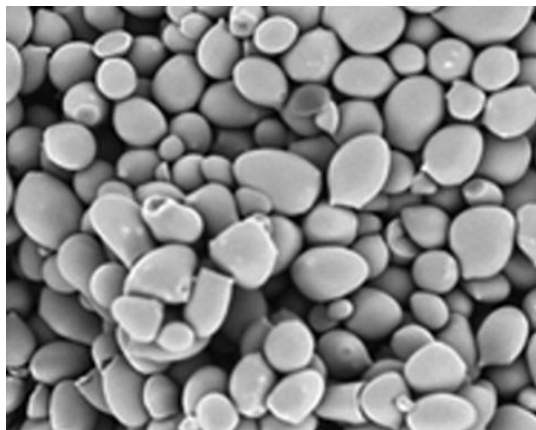


(a)

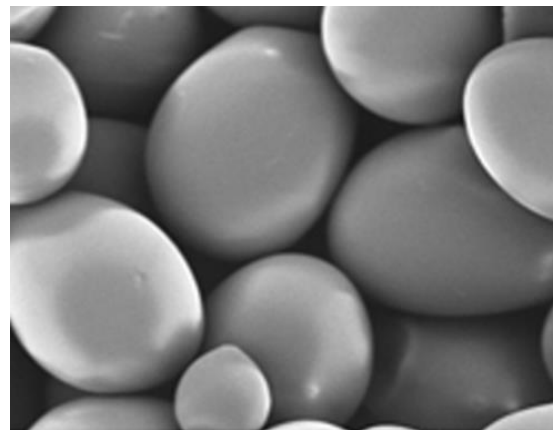


(b)

Figure 4.13: MC20 granules at (a) 500x magnification and (b) 2000x magnification



(a)



(b)

Figure 4.14: MC10 granules at (a) 500x magnification and (b) 2000x magnification

The appearances of each granule in all samples are varied as they consist of large, medium, and small broken granules that may have been generated as the effect of mechanical injury during extraction process. Figure 4.12 (a), 4.13 (a) and 4.14 show that all samples have similar shapes appearance. Since all samples were dried at similar temperature but different drying time to the extent of different final moisture content, the results indicate that drying time or final moisture content does not affect granule shapes. Meanwhile, Timm et

al. (2020) has mentioned that drying temperature may slightly change the starch shape. Furthermore, similar shapes and appearances also indicate that there were no changes in molecular structure during drying (Du et al., 2020).

SEM micrographs show starch granules with lower moisture content (MC20 and MC10) have smoother granule surface, while MC40 and MC30 showed some impurities at the surface. The impurities may be presumed as the bits of proteins and lipid which are also a significant properties of native sago starch (Othman et al. 2015). According to Aviara (2010), starch granules morphology is determined by the drying condition. Starch morphology also may affect the end-product quality such in baking product or in pasta (Patel & Seetharaman, 2006).

4.3.3 Effect of Drying on Sago Starch Granule Size

Figure 4.15 shows the particle size distribution of granules diameter between 0 to 15 μ m, while Figure 4.16 shows the dominant distribution of granules between 15 to 80 μ m.

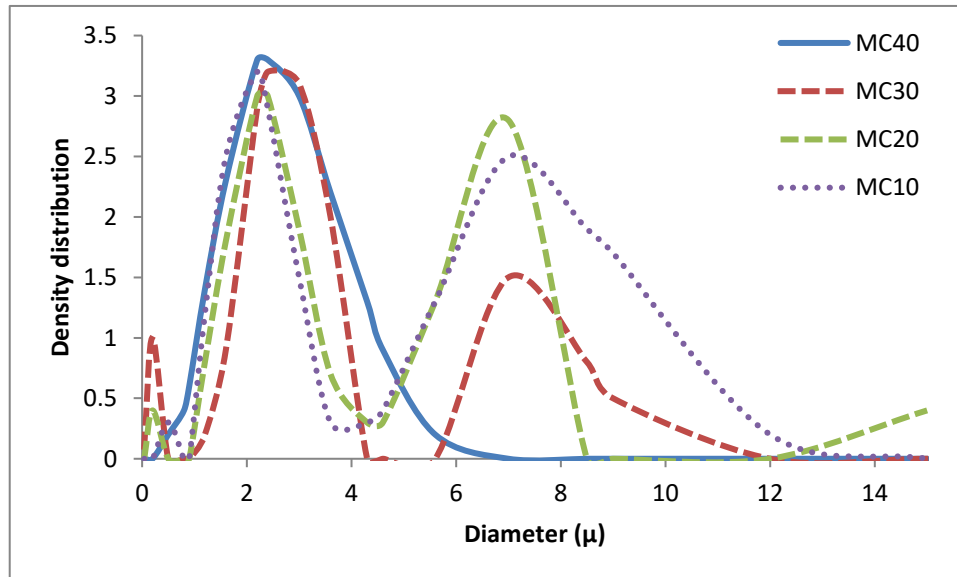
Apparently, all samples show tri-modal granule size distribution which was highly comprised between 30 to 50 μ m, with two slight shoulders at the low-end representing small granules or granules fragments at peak diameter of 3 and 7 μ m. In addition, the results indicate that the samples consisted of multiple size granules (larger, medium, and small) as shown in SEM results above. This broad range probably had been formed during the extraction process.

Meanwhile, Table 4.5 shows the mean diameter of sago starch granule in respect with their density distribution at 10%, 50% and 90%.

Table 4.5: Granule size distribution analysis of sago starch at different moisture contents

Sample	D10* ^c	D50* ^b	D90* ^a	Mean Diameter*
MC40	19.44 ± 0.13 ^c	29.80 ± 0.20 ^b	43.85 ± 0.16 ^d	30.14 ± 0.12 ^c
MC30	23.31 ± 0.27 ^a	35.59 ± 0.23 ^a	50.23 ± 0.22 ^b	35.10 ± 0.22 ^b
MC20	22.39 ± 0.10 ^b	35.34 ± 0.16 ^a	49.52 ± 0.21 ^c	34.83 ± 0.15 ^b
MC10	23.19 ± 0.13 ^a	35.59 ± 0.18 ^a	51.31 ± 0.16 ^a	35.67 ± 0.20 ^a

(*) All values are mean diameter of three replications. D10, D50 and D90 refer to the diameter in density distribution of 10, 50 and 90% of density distribution, respectively. Within the same pattern column and row (for D10, D50 and D90), the values with different letters are significantly difference at $p < 0.05$.

**Figure 4.15:** Particle size distribution of sago starch samples at different moisture content in wet basis for granule at diameter of 0 - 15µm

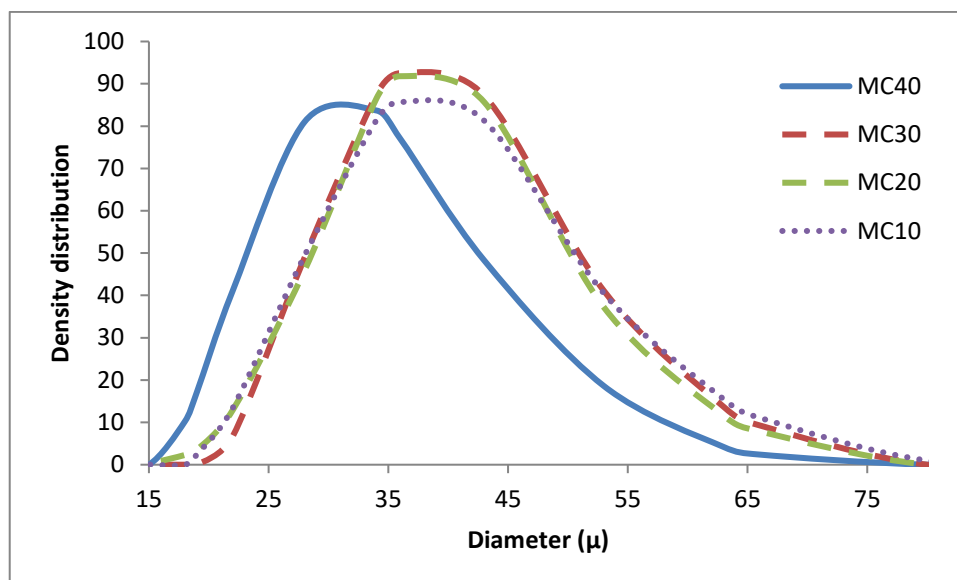


Figure 4.16: Particle size distribution of sago starch samples at different moisture content in wet basis for granule at diameter of 15 - 80µm

In all samples, D10 values range between 19.4 and 23.39µm; D50 between 29.80 and 35.59µm; D90 between 43.85 and 51.31µm; and the mean diameter ranges between 30.14 and 35.67µm. MC10 shows the highest mean diameter followed by MC20 and MC30. Rao and Tattiyakul (1999) also found that tapioca granule size increased upon longer drying times at a specific drying temperature, while Caparanga et al. (2017) found that arrowroot starch granule decreases upon increasing drying temperature but not air velocity. These findings indicate that granule size is not depending on moisture content. It can be influenced by increased of cavities at granule surface due to the drying process as suggested by Sujka and Jamroz (2006). However, statistical analysis results show that each parameter value varies significantly but in random way among all samples; suggested that the sago starch granules size are not affected by its moisture content.

The starch granule size is an important factor that will affect starch flow properties, apart from its density. Table 4.5 shows that D10, D50 and D90 value ranged between 19.4

to 23.39 μ m, 29.80 to 35.59 μ m; and 43.85 to 51.31 μ m, respectively. This broad variation of population may be formed during the extraction process. In addition, dried sago starch of MC30, MC20 and MC10 consists of a higher number of larger granules when compared to un-dried sago starch, which is probably due to agglomeration and initial swelling in dried material. Meanwhile, the mean diameter of sago starch granules for MC40, MC30, MC20 and MC10 were 30.14, 35.10, 34.83 and 35.67 μ m, respectively. MC10 showed the highest mean diameter, while MC40 showed the lowest mean diameter. Statistical results also showed that the mean diameter of MC30 is not significant with MC20 ($p>0.05$), but still differ significantly with MC10 and MC40 ($p<0.05$). Even though the result was not in increasing manner, the granule size for MC20 and MC10 were larger than granule in MC30 and MC40. This phenomenon is a typical phenomenon for starch granule in low temperature drying (below gelatinization temperature), which is called as reversible swelling (Alcázar-alay et al., 2015). However, earlier, Rao and Tattiyakul (1999) had discovered that tapioca granule size increased upon longer drying times at specific temperature but the moisture content was not observed. Meanwhile, Dai et al. (2008) had concluded that moisture content only creates minor effect towards granule size.

4.3.4 Effect of Drying on Sago Starch Color

Starch color is an important aspect for its general appearance and consumer acceptance. Figure 4.17 shows the color of sago starch in different moisture content. These samples look almost similar in colour, but they are in fact are different as indicated by the color measurements in Table 4.6.

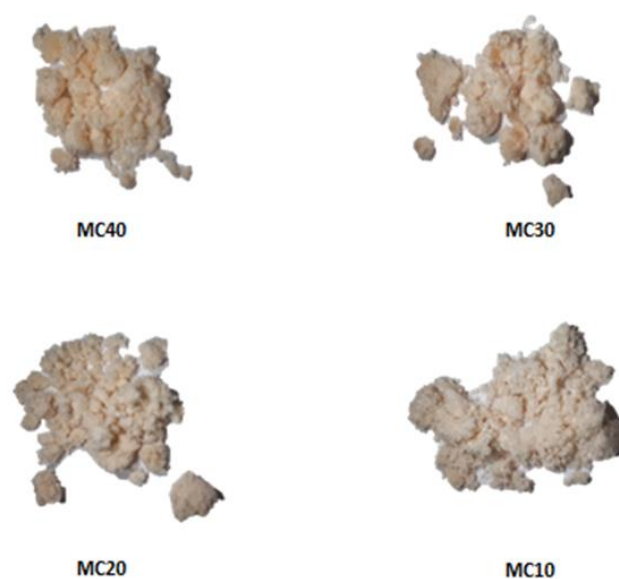


Figure 4.17: Color analysis of sago starch samples at different moisture content

Table 4.6: Color analysis for Hunter L*, a*, b* and whiteness index of sago starch at different moisture content

SAMPLE	L*	a*	b*	Whiteness*
MC40	61.35 ± 0.38 ^d	15.35 ± 0.05 ^d	9.65 ± 0.02 ^d	57.67 ± 0.02 ^d
MC30	79.31 ± 0.09 ^c	3.23 ± 0.14 ^c	8.72 ± 0.01 ^c	77.41 ± 0.02 ^c
MC20	82.49 ± 0.22 ^b	1.24 ± 0.01 ^b	6.78 ± 0.01 ^b	81.41 ± 0.01 ^b
MC10	85.49 ± 0.22 ^a	0.04 ± 0.01 ^a	5.12 ± 0.01 ^a	84.71 ± 0.34 ^a

*Reported values are the mean ± Standard Deviation, SD (n=3). The lower-case letters indicate significant differences ($p \leq 0.05$) within the same row, generated by ANOVA

Figure 4.17 apparently shows that all samples have similar color appearance. However, image analysis results for color measurement show that each of the sample has different color value. L* value indicates lightness, (+ve) a* value indicates red color, (-ve) a* indicates green color, (+ve) b* value indicates yellow color, (-ve) b* value indicates blue color and WI is whiteness index. The Hunter L* values for the samples at Table 4.6 were found to be between 61.35 and 85.73. MC40 with the highest moisture content has the lowest L value of 61.35, followed by MC30 (79.31), MC20 (82.49) and MC10 (85.49). L value has positive correlation with Whiteness Index (WI). Therefore, WI also shows similar trend that is the value increased from MC40 to MC10. The WI for MC40, MC30, MC20 and MC10 are 57.67, 77.41, 81.41, and 84.71, respectively. This indicates that sago starch color getting whiter in drier condition. The statistical result also shows that final moisture content affect sago starch color properties.

Table 4.6 also shows that Hunter a* values for MC40, MC30, MC20 and MC10 are 15.35, 3.23, 1.24, and 0.04, respectively. Meanwhile the Hunter b* values for MC40, MC30, MC20 and MC10 are 9.65, 8.72, 6.78, and 5.12, respectively. The results for Hunter a* and b* show that both values decreased in sample with lower moisture content. Moreover, the statistical results of lower alphabet case indicates that Hunter a* and b* value were significantly affected by final moisture content. The results also in negative correlation with Hunter L* and WI value. Hunter (+ve) a* and (+ve) b* value is related with red and yellow, respectively. Therefore, high value of Hunter (+ve) a* and (+ve) b* in MC40 and MC30 may result in pinkish appearance as can be seen in Figure 4.17.

The pinkish color of sago starch is the effect of browning which probably come from the presence of polyphenol-oxidase activity. The presence of polyphenol comes from affected ecosystem around the origin palm, as suggested by Konuma et al. (2012) who had

conducted study about the relation between starch browning and their ecosystem. However, polyphenols may dissolve in water. During drying, polyphenols will disappear together with the moisture loss resulting in whiter colour, as observed in MC20 and MC10. Alean et al. (2016) also had documented the polyphenol kinetic during cocoa drying that also decreased according to moisture content and drying time. That is the reason why the whiteness is different for different drying temperature.

The L*a*b* and WI value was plotted against moisture content and the graphs are presented as in Figure 4.18 to Figure 4.21 below.

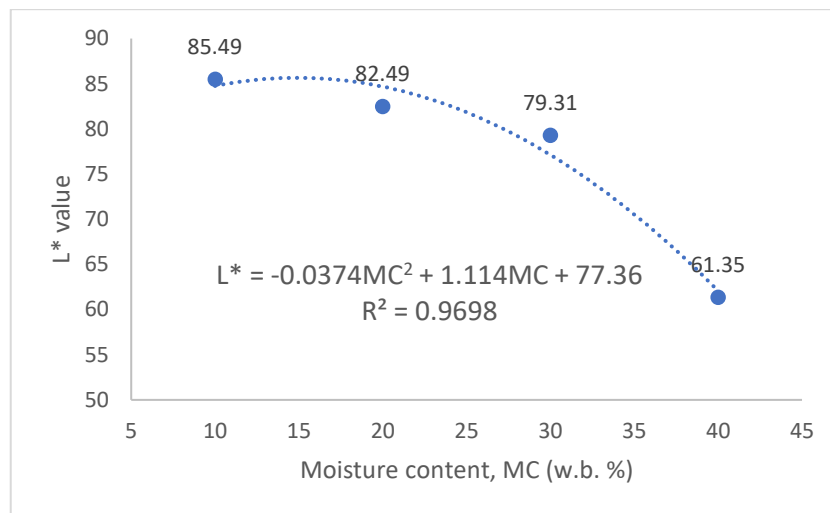


Figure 4.18: L value vs Moisture content, MC (w.b. %)

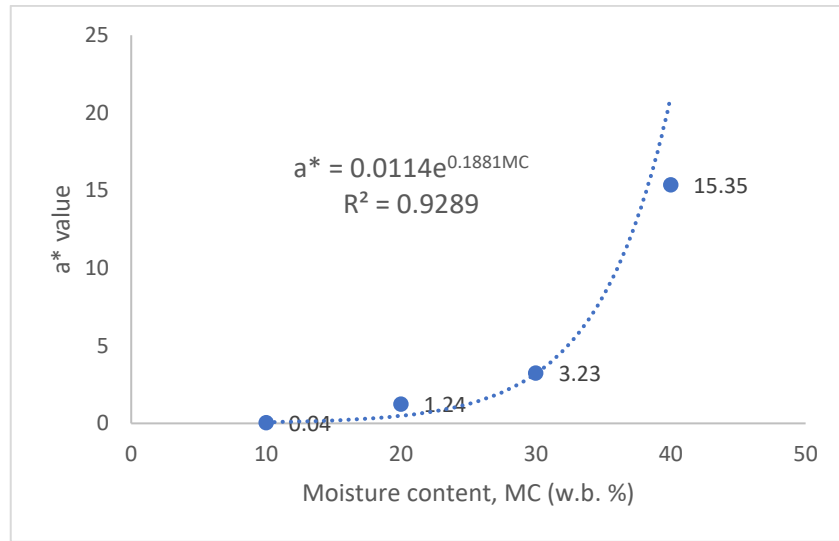


Figure 4.19: a value vs Moisture content, MC (w.b. %)

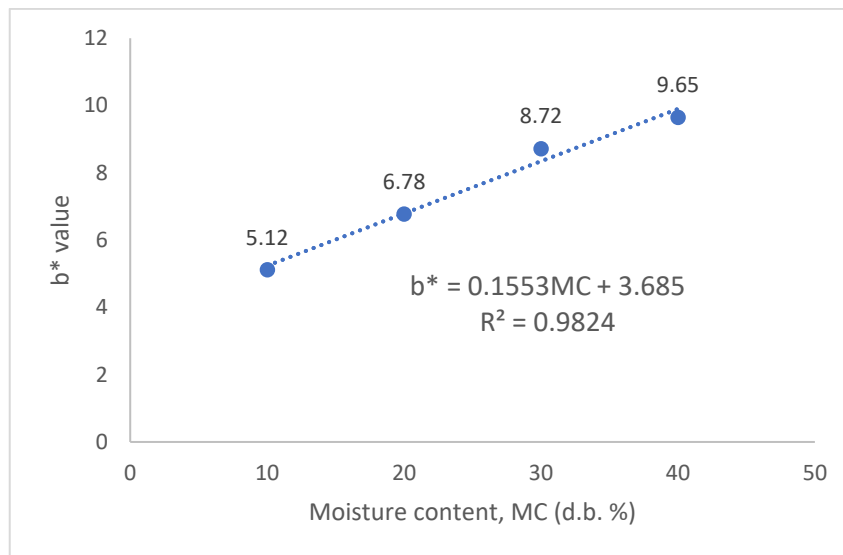


Figure 4.20: b value vs Moisture content, MC (w.b. %)

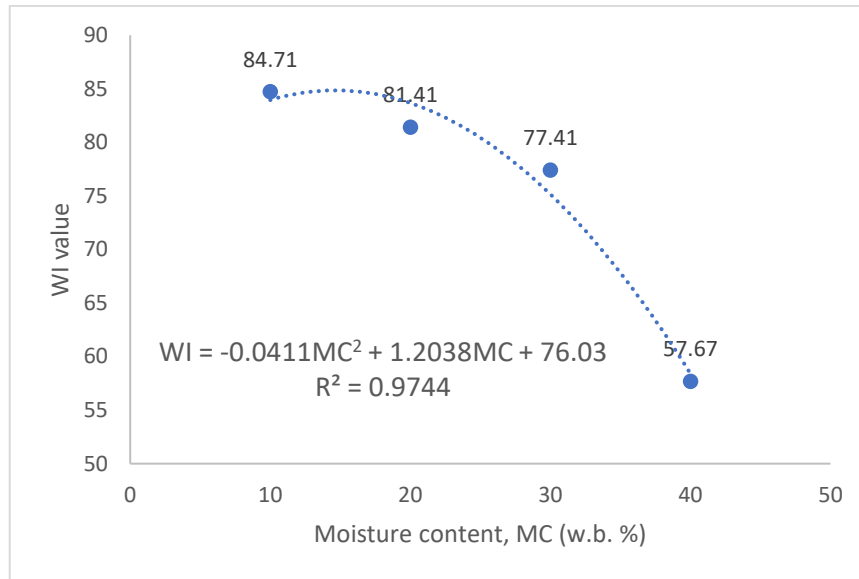


Figure 4.21: WI value vs Moisture content, MC (w.b. %)

The dependence of color properties with moisture content (MC) could be represented by the following expression:

$$L = -0.037MC^2 + 1.114MC + 77.36 \quad \text{Equation 4.2}$$

$$a = 0.011e^{0.18MC} \quad \text{Equation 4.3}$$

$$b = 0.15MC + 3.68 \quad \text{Equation 4.4}$$

$$WI = -0.041MC^2 + 1.204MC + 76.03 \quad \text{Equation 4.5}$$

Where MC is the moisture content in dry base (%) with corresponding R² values of 0.9698, 0.9959, 0.9824 and 0.9744, respectively.

4.3.5 Effect of Drying on Sago Starch Peak Viscosity

Brabender Amylograph was used to observe the pasting characteristic of sago starch at different moisture content. Table 4.7 presents the Peak Viscosity of dried sago starch. Native sago starch is known as good quality high-viscous starch and high thickening power;

therefore, MC40 indicated the highest PV value that was 659, followed by MC30, MC 20 and MC 10 that were 621, 607 and 575, respectively (Wattanachant et al., 2002).

Table 4.7: Peak Viscosity of sago starch at different moisture content

SAMPLE	Peak Viscosity, PV
MC40	659 ± 1.00 _d
MC30	623 ± 2.08 _c
MC20	607 ± 6.00 _b
MC10	574 ± 4.16 _a

*Reported values are the mean ± Standard Deviation, SD (n=3). The lower-case letters indicate significant differences ($p \leq 0.05$) within the same row, generated by ANOVA

Statistical result also showed that the peak viscosity was significantly increased ($p < 0.05$) when the moisture content increased (Appendix: Table 5). The peak viscosity (AU) was plotted against moisture content and the graphs are presented as in Figure 4.22 below.

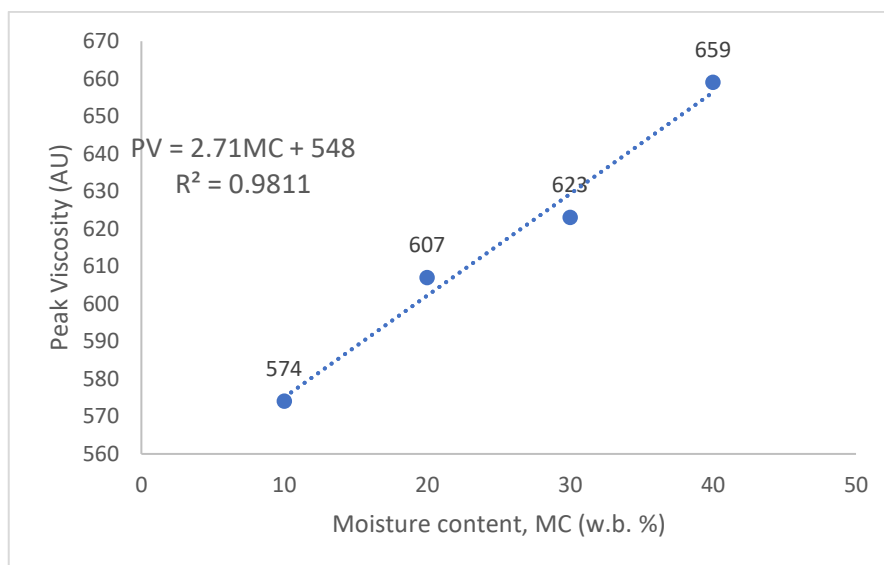


Figure 4.22: Peak viscosity of sago starch at different moisture content

The relationship between moisture content and peak viscosity (PV) can be represented by the following equation:

$$PV = 2.71MC + 548 \quad \text{Equation 4.6}$$

With R^2 value of 0.9811.

Figure 4.22 shows that peak viscosity increased in sample with more moisture content. It means that dried samples have less viscosity compared with native samples. Native sago starch has stronger molecular bond that is not easily disrupted during heating; thus, give highest value for peak viscosity. Meanwhile, among the dried sago starch, sample with lower moisture content showed lower peak viscosity probably due to the presence of amylose outside the granules as the results of amylose leaching during drying and swelling (Dehnad et al., 2016).

4.3.6 Effect of Drying on Sago Starch Molecular Characterization

In this study FT-IR was used to evaluate compound differences between treated starches and their spectra were recorded as shown in Figure 4.23 below.

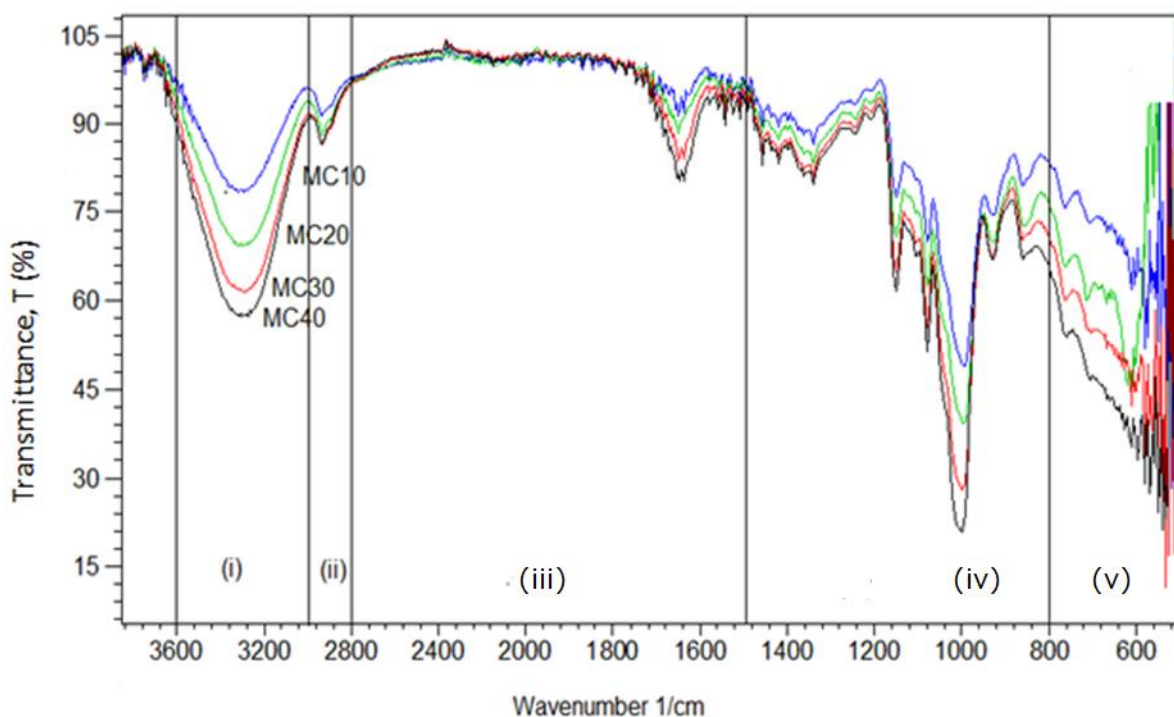


Figure 4.23: FT-IR spectra of four different sago starch samples treated to different moisture content labelled as MC40, MC30, MC20 and MC10. The roman numeral labels refer to FT-IR main region for starch (i) O-H stretch; (ii) C-H stretch; (iii) H stretch; (iv) fingerprint of glycosidic linkages; (v) skeletal mode of pyranose rings (Kizil et al., 2002)

The spectra were classified into five regions as labelled in roman numerical. Region (i) of peak at 3000 to 3600 cm^{-1} is referring to O-H stretch; region (ii) of peak at 2800 to 3000 cm^{-1} is referred to C-H stretch, region (iii) of peak at 1600 cm^{-1} is referred to H stretch, region (iv) of peaks at 1000 cm^{-1} is referred to glycosidic linkages; and lastly region at (v) is

500 to 800 is referred to skeletal mode of pyranose rings. The combination of all peaks especially the strong and medium showed the carbohydrate nature of the starch samples.

The patterns of all spectra obtained were similar but different in their intensities with some peaks slightly shifted which attributed to the effect of their moisture content. The patterns similarity had strongly indicated their identical vibrational modes of amylose and amylopectin among all samples. Besides, it also means that drying treatment had changed neither the type of chemical group nor produced new chemical group in starch molecules (Braşoveanu et al., 2013). Furthermore, difference in intensities can be observed in almost all strong and medium peaks. The most significant intensities can be observed at peak 3302 cm^{-1} in O-H stretching region (3000-3600 cm^{-1}) of hydroxyl group. The peak is highest in MC40 and lowest in MC10 perhaps related to the amount of water molecule in each moisture content. Region (ii) has only one peak of C-H stretch which shows different intensities due to the variations of amylose and amylopectin amount in samples (Kizil et al., 2002). The peaks at 1077 and 1151 are belong to the C-O-C and C-O bond of the glycosidic linkages in starch in which decreasing in their intensities showed that drying had resulted in breakage of glycosidic linkages in starch that will affect its crystallinity. The low wavenumber region below 800 cm^{-1} shows complex vibrational modes due to skeletal modes vibration of pyranose ring in glycosidic unit. Moreover, the peaks at 1642 cm^{-1} also exhibited difference intensities attributed to the amount of water adsorbed in amorphous region which affected starch crystallinity (Kizil et al., 2002).

4.3.7 Effect of Drying on Sago Starch Thermal Stability

In this study, TGA had been used to evaluate the thermal stability of sago starch in different moisture content and the thermogram is shown in Figure 4.24. All samples similarly showed similar TGA pattern of four phase. The first phase is between 0 to 150°C; the second phase is between 150 to 290 °C; the third phase is between 290 to 320 °C; and the last phase is from 320 to 600 °C. It also can be seen that sago starch mass decreased significantly in the first phase, became constant at the second phase, decreased significantly again at the third phase; but decreased gradually at the final stage.

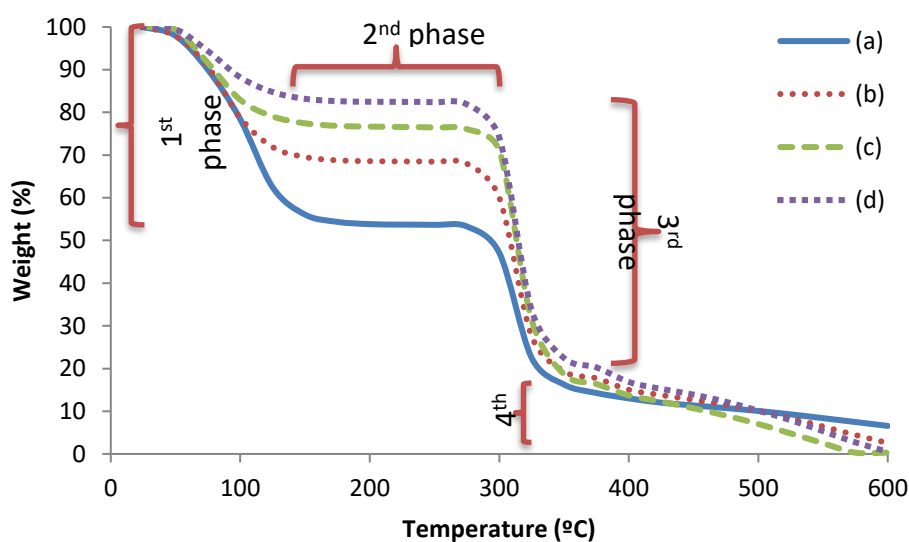


Figure 4.24: TGA of sago starch at different moisture content.

(a) MC40 (b) MC30 (c) MC20 (d) MC10

In the first phase, MC40 shows the steeper curve, followed by MC30, MC20 and MC10. This indicates that MC40 increasingly loses more water, followed by MC30, MC20 and MC10. This is because the mass loss in first phase is related with the evaporation of bound water in both crystallite and amorphous region (Zhiqiang et al., 1999). In other words,

sago starch with lower moisture content has more thermal stability at temperature below 150 °C as less evaporation occurred around its molecules.

In the second phase, as the evaporation process is completed, all sago starch similarly shows moderate thermal stability up to 300 °C that is not depending on amount of water. This characteristic is a desirable property in the food industry which is useful for further industrial application.

The third phase of weight loss occurred faster with steeper curves resulting in maximum weight loss up to 59.6% for MC10, 52.4% for MC20, 49.1% for MC30 and 32.7% for MC40. MC40 shows lowest weight loss which clearly indicates that native starch has the most thermal stability to hold its crystalline structures, as compared to other samples. This is because, this phase is the decomposition stage which corresponds to the dehydration of saccharide rings and subsequently de-polymerization of starch macromolecules (Piyada et al., 2013).

Finally, the char yield or ash (resides) varies randomly among the MC40, MC30, MC20 and MC10 which are 6.5%, 0.3%, 2.5% and 0.4%, respectively. The results indicate that the char yield is not affected by sago starch moisture content. It has been suggested that the char yield is influenced by the plantation or processing condition (Mustafa Kamal et al., 2007).

4.3.8 Concluding Remarks

The results obtained from this section show that different moisture content cause changes in some of the sago starch properties such as color, peak viscosity, molecular characterization, and thermal stability. This indicates the importance of moisture content

consistency in sago starch experiment and processing. The next subtopic will be discussing how drying variables and parameter affect the result of final moisture content.

4.4 Effect of Drying on Sago Starch Final Moisture Content

4.4.1 Effect of Drying Temperature on Sago Starch Final Moisture Content

The equilibrium moisture content or EMC is termed as the moisture content of a wet solid in equilibrium with air of given humidity and temperature. As for drying, EMC is a state when moisture movement becomes very slow, the moisture content changes become negligible, and the drying can be considered completed, normally after long drying time (depending on drying condition). In a drying curve profile, the EMC state is usually recognized as a long-stable-horizontal line at the last drying stage. The EMC of sago starch dried in different temperatures (50, 60, 70 and 80°C) are shown in Figure 4.25.

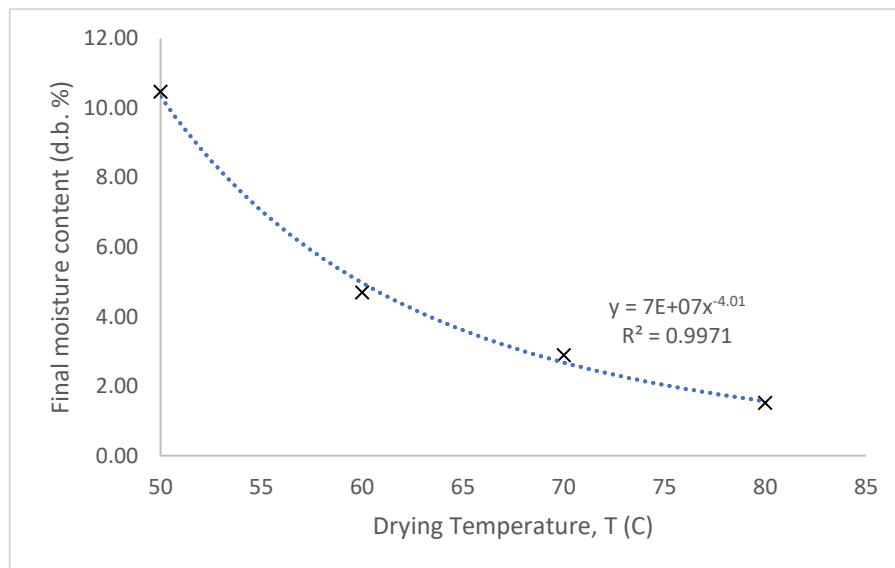


Figure 4.25: Final moisture content of sago starch at different drying temperature

The relationship between EMC and drying temperature can be represented by the following equation:

$$EMC = 7 \times 10^{7T^{-4.01}} \quad \text{Equation 4.7}$$

The value of R^2 is 0.997. $50^\circ\text{C} \leq T \leq 80^\circ\text{C}$.

Where EMC is the final equilibrium moisture content (%(d.b.)), while T is the drying temperature ($^\circ\text{C}$). In contrast, Aviara and Igbeka, (2016) had found linear relationship between EMC and drying temperature for cassava for a range of temperature between 40 to 60 $^\circ\text{C}$, which also suggest that different material has different relationship. The different relationship between EMC and temperature found for different materials may be due to the difference in the internal structure of material, which becomes the hindrance factor to moisture movement.

The EMC value is influenced by hygroscopic tendencies of sago starch to hold moisture. Sago starch dried at 50°C shows the highest EMC at 10.47%, followed by 60 $^\circ\text{C}$ at 4.70%, 70 $^\circ\text{C}$ at 2.89%, and 80°C at 1.52% as the lowest EMC. The result suggests that drying temperature has an influence towards the hygroscopic properties due to the changes in hydroxyl group. In certain starch usages such as baking cakes, its hygroscopic property is important to absorb moisture during food process. In terms of cost effectiveness, drying at low temperature can be a good option due to less energy used and better quality of dried product. However, the final moisture content of material dried at lower temperature usually higher than those obtained for material dried at higher temperature. Similar results of decreasing EMC upon increased drying temperature were also found by Diamante et al. (2010) for kiwi fruit slices dried around 60 to 100°C .

The EMC of sago starch dried at 50°C was two-times higher than the EMC of sago starch dried at 60°C, three-times higher than the EMC of sago starch dried at 70°C, and six-times higher than the EMC of sago starch dried at 80°C. The result shows that the gap in the final moisture content between the lowest and highest temperatures is significant.

The value of EMC decreased exponentially with the increase in drying temperature within a temperature range of 50 to 80°C indicating the significant influence of heating to the moisture content reduction. The drastic change of EMC is also associated with the drastic change of relative humidity due to the increase in temperature in the drying oven, which has been studied by Kolawole (2013) who study the relationship between drying temperature and humidity within an oven.

4.4.2 Effect of Air Velocity on Sago Starch Final Moisture Content

The plot of air velocity versus EMC is shown in Figure 4.26.

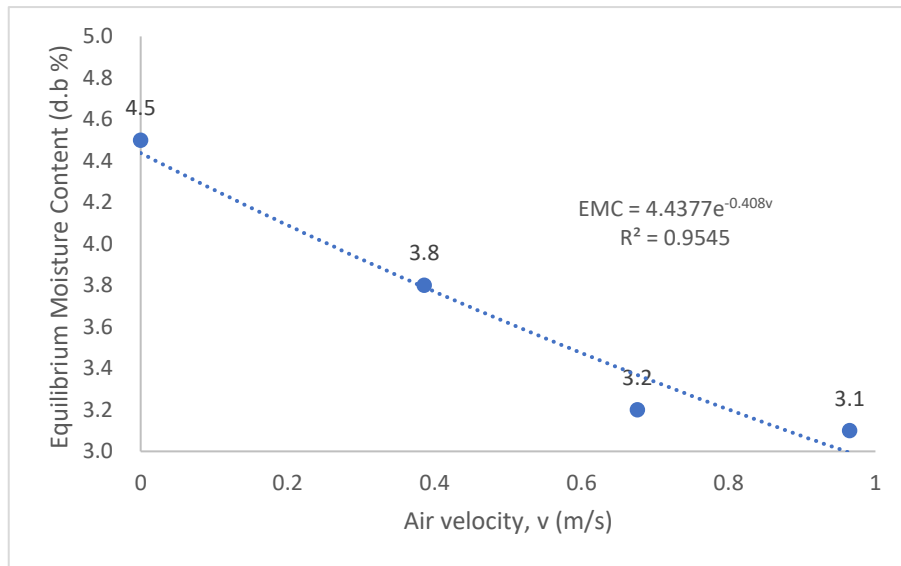


Figure 4.26: Equilibrium Moisture Content of sago starch at different air velocity

The value of EMC decreased exponentially with the increase in drying temperature within air velocity range of 0 to 0.965 m/s. The slow changes of EMC might be influenced by the slow changes of relative humidity in the drying oven. Air flow acts as a medium to accelerate moisture movement which directly influenced oven humidity. However, the results obtained showed that the air velocity has less effect to decrease the EMC during drying as compared with temperature effect. The relationship between EMC and air velocity can be expressed with the following equation.

$$EMC = 4.4377e^{-0.408v} \quad \text{Equation 4.8}$$

$$R^2=0.9545; 0 \leq v \leq 0.9545 \text{ m/s}$$

The EMC for sago starch dried found were 4.5, 3.8, 3.2 and 3.1 d.b. %, for velocities of 0, 0.386, 0.676, and, 0.965 m/s, respectively. The result shows that the faster the air velocity, the lower the EMC recorded because higher air flow transferred higher amount of moisture. However, the effect of air velocity is found to be less compared to the effect of temperature on the EMC. The EMC was reduced by 31% when the air velocity increased from 0 to 0.965 m/s with constant temperature of 60°C while it was reduced by 50% when temperature was increased from 60°C to 80°C with constant air velocity. This shows that drying temperature has more influence towards EMC as compared with air velocity. Similar results were also found by Caparanga et al. (2017) for arrowroot starch dried at 65 °C with air velocity range from 0.4 to 0.6 m/s which suggested less effect from air velocity.

4.4.3 Effect of Sample Thickness on Sago Starch Final Moisture Content

In this experiment, three sago starch sample with different thickness were subjected to drying 60°C and air velocity of 0.386 m/s for 11 hours. The parameter of sample thickness was 0.5, 1 and 2 cm. Figure 4.30 shows the effect of sample thickness during drying to the EMC of sago starch.

Figure 4.27 shows that sago starch dried at 0.5 cm thickness shows the lowest EMC at 3.805% d.b, followed by 1 cm at 4.833% d.b, and 2 cm at 18.039% d.b. It deducts that the EMC increases as the sample becomes thicker, which means a positive correlation between both parameter. This is in contrast with the results obtained from the effect of temperature and air velocity in previous sections. The result shows that sample thickness has a significant effect on EMC which is more obvious at thicker extent. When the thickness was increased two times (from 1 cm to 2 cm), EMC increased nearly 4 times, as shown in Figure 4.27. The increase in EMC for the increase in thickness highlights the increase hindrance moisture loss caused by the higher mass, which contains more both of solid and moisture content. This can be influenced by the differences of bound water in the initial sample.

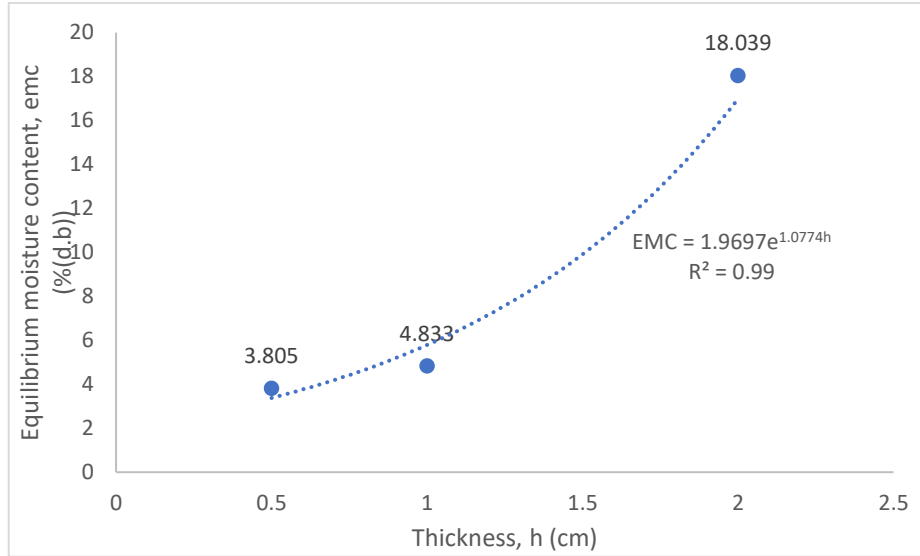


Figure 4.27: Equilibrium Moisture Content at different sample thickness

EMC is related to water activity and consequently water content of a sample. Water content plays a major role in determining materials water activity involving insects' survival, microorganism productivity and enzyme activity which leads to products shelf life. Though drying at 2 cm thickness increased the EMC, the figure is still below 18% w.b. which is safe to prevent microbial activity.

$$EMC = 1.97e^{-1.0774h} \quad \text{Equation 4.9}$$

$$R^2=0.99; 0.5\text{cm} \leq h \leq 2\text{cm}$$

Where EMC is the equilibrium moisture content, EMC (d.b), while h is the sample thickness, h (cm).

4.4.4 Effect of Sample Position on Sago Starch Final Moisture Content

In this experiment, three sago starch sample with different positions inside an oven were subjected to 60°C drying at air velocity of 0.386 m/s for 12 hours. The calculation for air velocity is shown in Appendix. All samples were dried until they reach equilibrium state. The mass of the samples was recorded to calculate their equilibrium moisture content. The result is presented in Figure 4.28 below.

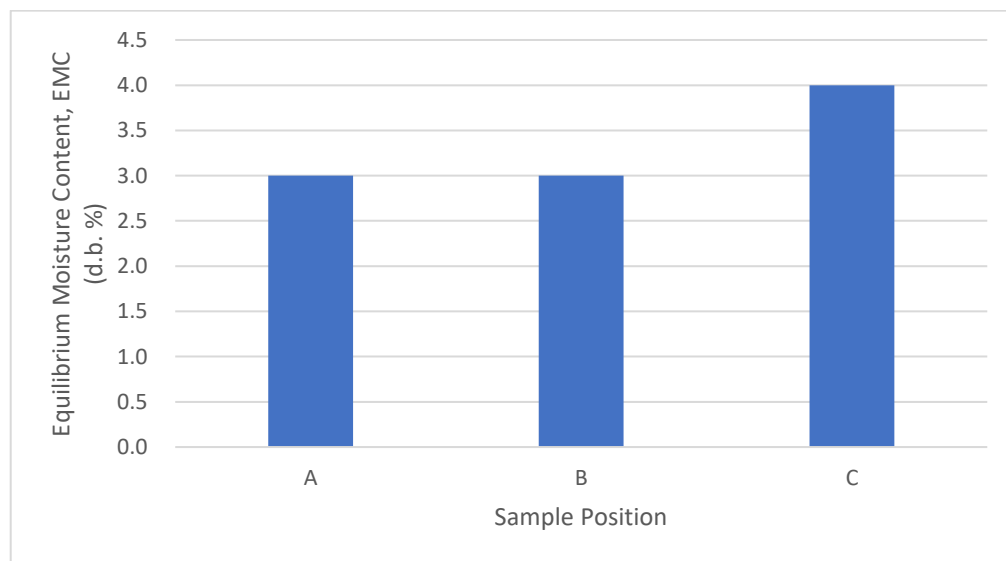


Figure 4.28: Equilibrium moisture content at different sample position A is referred to low level-side position; B is referred to low level-middle position; and C is referred to top level-middle position in a natural convection drying oven.

Figure 4.28 shows that sago starch placed above fan in an oven shows the highest EMC at 3.8% d.b, while samples A and B which were placed at the same height with fan show similar EMC that is 3.3% d.b. This result shows an insignificant increase in EMC, about 13%, when the height level is raised by 50%. This indicates the insignificant effect of location on the EMC.

Even though EMC between sample C is different with sample B and C; however, according to t-test, the EMC value between all samples were insignificant ($p>0.05$). Similar results has been found by Bradley (2010) when dried cheese slices vertically and horizontally inside a forced draft oven.

4.4.5 Effect of Drying Method on Sago Starch Final Moisture Content

In this experiment, sago starch samples were subjected to various drying methods that is oven (continuous), oven (intermittent), sun drying, microwave and freeze drying. The equilibrium moisture content, EMC of the samples at the end of drying experiment were recorded and presented in Figure 4.29 below.

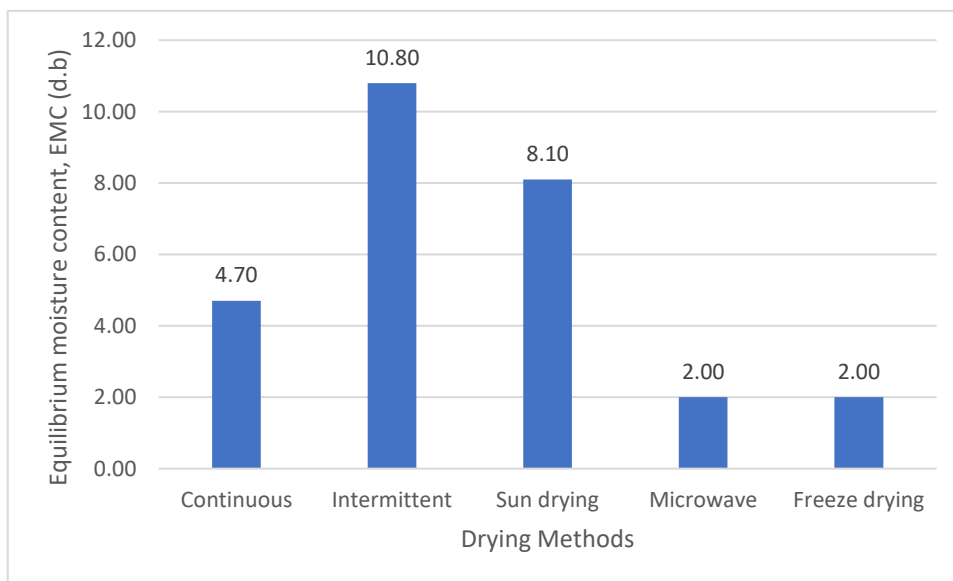


Figure 4.19: EMC of samples at different drying methods

Figure 4.29 depicts that sago starch dried through intermittent drying method has the highest final moisture content that is 10.8% d.b, followed by sun drying that is 8.10 % d.b. This result shows the advantage of using intermittent drying compared to other drying

techniques. For the intermittent drying, drying was done continuously for one hour. Then, the heating was turned off for 1 hour, and a similar schedule was used until the equilibrium moisture content was reached. The off mode of drying for intermittent drying reduces the energy used as well as drying time. Drying was fast during the continuous drying period, and it stopped during the off-heating period, which allows the moisture to travel from the internal part of the material to the surface. The accumulated moisture at surface resulting to the increase drying rate during the subsequence continuous drying period. Meanwhile, sun drying is known as natural intermittent drying. Sago starch dried through sun drying has more than double final MC that is 8.10% d.b as compared to oven-intermittent drying. This result might be influenced by outdoor humidity in Malaysia which is around 80% on average. On the other side, the humidity inside the drying oven was determined as 12% for 60°C drying temperature.

Microwave and freeze-drying resulted in sago starch with the lowest EMC that are 2.0% d.b. This indicates that radiation process from microwave drying, and sublimation process from freeze drying are the most powerful methods to produce driest product. However, it is noted that the highest moisture content allowed for food product to avoid microbial activity is 15% d.b as referred to Malaysia Standard.

4.4.6 Concluding Remarks

The results from this section show that drying method, temperature, air velocity, and sample thickness affect the final moisture content of sago starch. This indicates that the adjustment of these variables is needed to obtain different final moisture content. In addition, correlation between final moisture content with sago starch properties has been discussed in previous section.

4.5 Drying Kinetic of Sago Starch

4.5.1 Effect of Drying Temperature to Sago Starch Drying Kinetic

Figure 4.30 shows sago starch drying profiles presented as moisture content versus time for temperatures of 50, 60, 70 and 80°C.

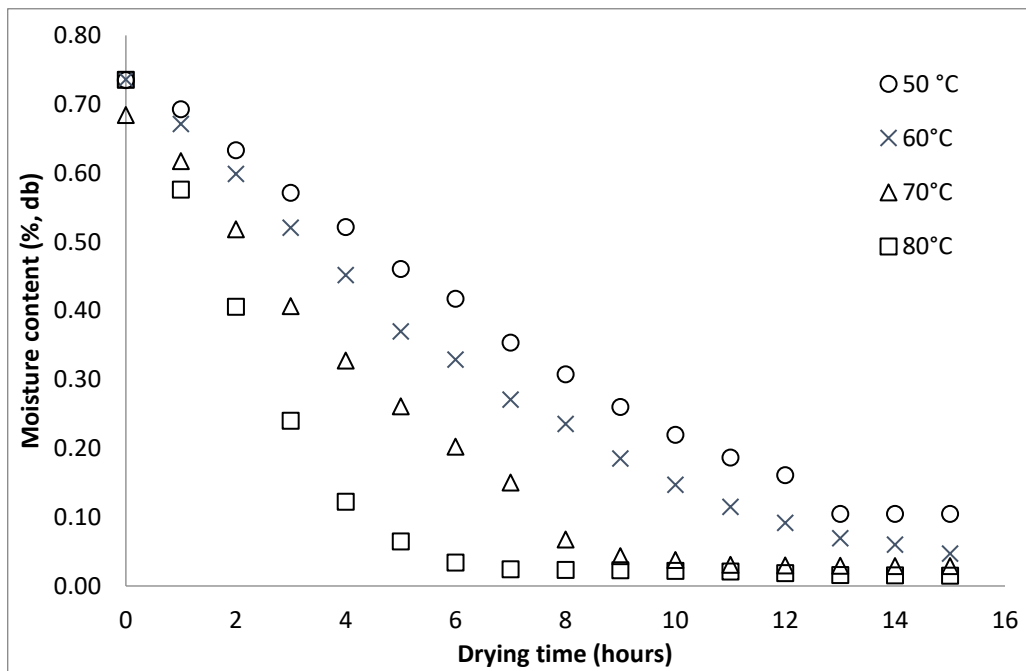


Figure 4.30: Sago starch moisture content at different drying temperature

In general, the kinetic of moisture loss was characterized as gradual decreasing in the first phase, with stabilization occurred at the final stage of drying process. Figure 4.30 shows that moisture content of all sago starch samples decreased upon drying time at all drying temperature. This indicates the effect of heating on the moisture loss of sago starch. In the early stage of drying, the curves are steeper since the moisture reduction mostly involves the moisture on the surface which can evaporate easily. In the later stage, the moisture decreased almost linearly at all temperature as evaporation occurred slowly due to the small amount of

moisture on the surface. Sample dried at 80 °C has the steepest curve at the first phase which also resulted in early accomplishment of drying process after six hours. The curve gets more moderate at lower drying temperature as well as the drying time getting longer. The drying time for 70°C ended at ten hours, while both the drying time for 60 and 50 °C are 15 hours. This indicates, higher temperature can save drying time, while drying at 50 and 60 °C does not have effect on drying time. Further explanation on drying temperature effects on drying kinetic is discussed through drying rate results in Figure 4.31.

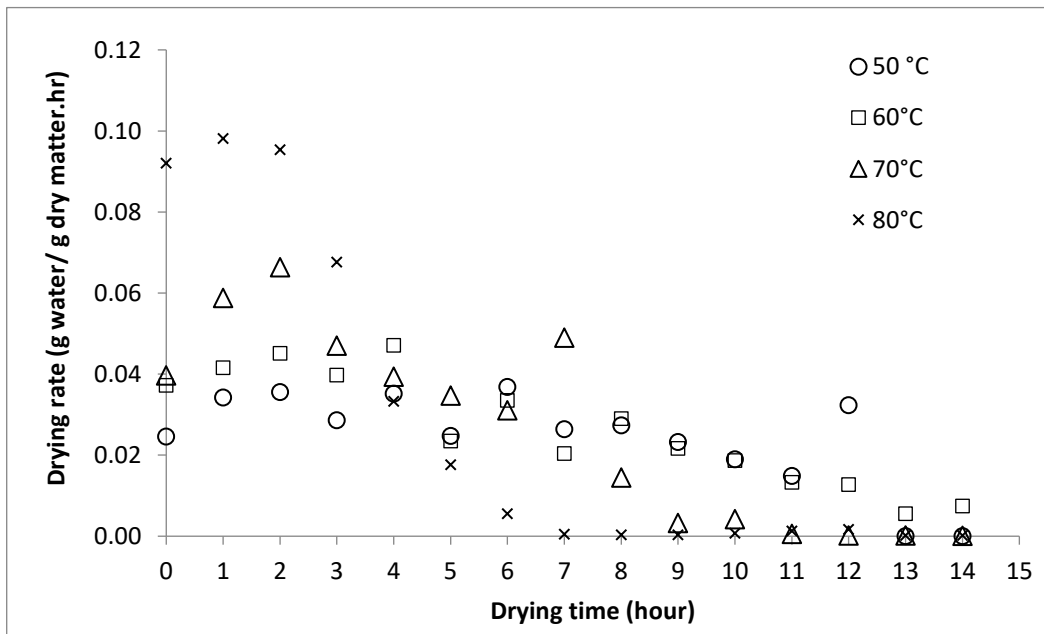


Figure 4.31: Sago starch drying rate at different drying temperature

Figure 4.31 shows that at all drying temperature drying rate is higher in the initial phase as compared to later stage. The result indicates moisture loss occurred more rapidly at the initial phase as compared to later stage. At the initial phase, the drying rate was high due to rapid moisture transfer as free moisture accumulates mostly on the surface of drying material, resulting to easy escape of moisture per unit time in the atmosphere. At the later

stage of all drying temperature, moisture mostly exists within the material and the cell matrix within the material hindered the drying resulting to slow drying rate. It is also noted that the drying rate increased to the highest rate after two or three hours before decreasing gradually. This indicates the time needed to transfer the heat into the sample, as well as to break the hydroxyl bond within.

Furthermore, at the early stage, the drying rate at 80°C was 40% higher than the drying rate at 50°C. This is because higher drying temperature caused higher heat to be transferred into the starch samples, resulting in significant effects on the amount of moisture removal. After four hours, the drying rate at 80°C decreased significantly followed by 70°C, 60°C and 50°. The decreasing trend of drying rate may be caused by the material internal structure and probably due to the formation of surface hard layer as the effect of rapid surface evaporation at the early stage of drying. This result is similar with those findings found for other food drying, and among those is that presented by Thao and Noomhorm (2011) who suggested that the falling rate found for sweet potato in their work was due to the increase in resistance to heat and mass transfer inside material. Low drying rate observed for low temperatures has been found to relate with the lesser effect on the quality of the dried material, and among the findings is from Gulati and Datta (2015) who suggested that drying at 50°C and 60°C can prevent the formation of surface hardening. Finally, at the end of the experiment, the drying rate of all samples were significantly low, as less moisture available within the sample meaning less moisture loss per unit time. Therefore, it is concluded that in the case of temperature, drying at 80°C is the most optimum drying temperature.

4.5.2 Effect of Air Velocity to Sago Starch Drying Kinetic

Figure 4.32 shows the sago starch drying curves of moisture content versus time for different fan settings of 0, 40, 70 and 100% which giving air velocity of 0, 0.386, 0.676 and, 0.965 m/s, respectively.

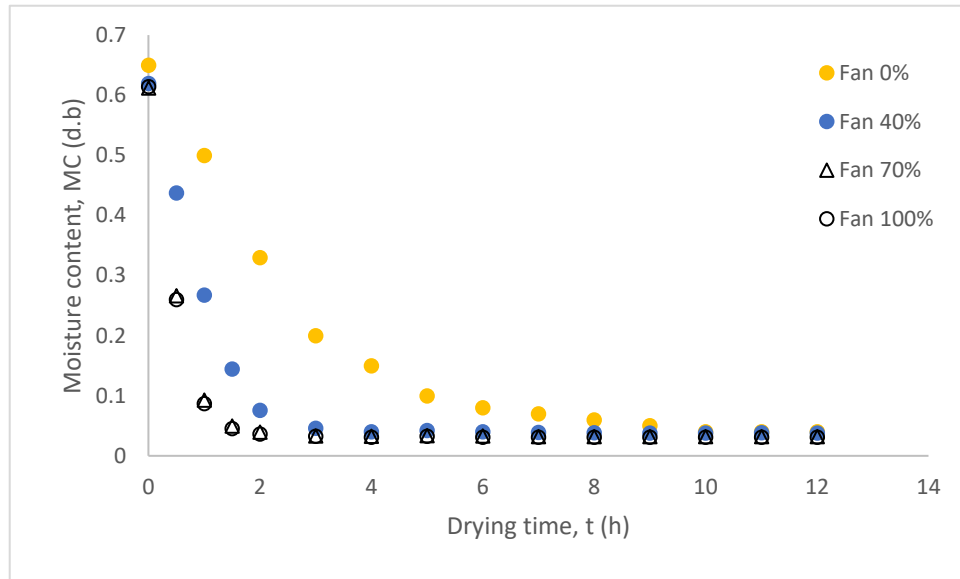


Figure 4.32: Moisture content at different fan setting

Figure 4.32 shows that the moisture content for all samples decreased with the increase of drying time at all air velocity. The sago starch dried at higher air velocity dried faster than sago starch dried at lower air velocity. It took around 12 hours for sago starch dried at 0% fan speed to reach equilibrium moisture content (EMC), while only 3 hours for sago starch dried at 100% fan speed. This indicates that the introduction of air velocity had decreased the drying time by up to three quarter hours. It is expected that rapid air movement around the sample has helped the moisture to escape from the surface to evaporate to the air. Further explanation on the effect of air velocity on sago starch drying kinetic is shown in Figure 4.33 below.

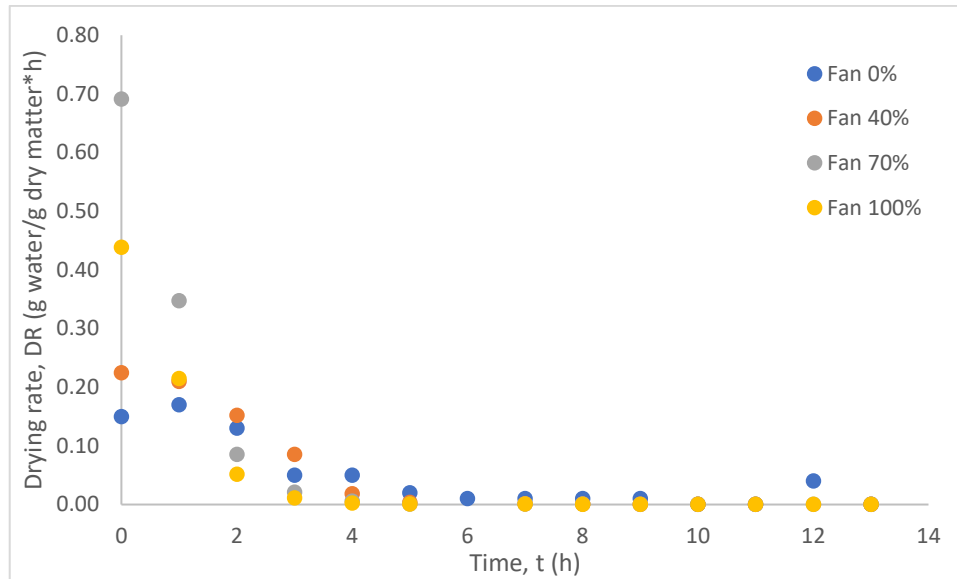


Figure 4.33: Drying rate at different fan setting

Figure 4.33 shows that the initial drying rate of sago starch dried in 70% fan speed was 93% higher than that of 0 air velocity. Interestingly, the drying rate at 70% fan speed was higher and has a steeper curve than that of 100% fan speed. The results suggest that air velocity assists drying process to increase moisture evaporation on the surface to a certain extent. This result is similar with the result from Maskan et al. (2002) who concluded that the air velocity in the drying of pestil can be neglected. Pestil is fruit juice concentrate-starch mixture. In conclusion, the results explained that air velocity can be combined with drying temperature as a drying coefficient to increase heat and transfer process.

4.5.3 Effect of Sample Thickness to Sago Starch Drying Kinetic

Figure 4.34 shows sago starch drying curves of moisture content versus time for different sample thickness of 0.5 cm, 1cm and 2 cm. Figure 4.34 shows that moisture content decreases along with drying time at all sample thickness. However, the curve for 0.5 cm sample thickness is the steepest, followed by 1 cm and 2 cm sample thickness. This indicates

that the moisture loss in 0.5 cm sample thickness was the fastest in which the moisture content dropped from 0.72 to 0.12% d.b within the early 3 hours. Meanwhile 1 cm sample thickness took four hours to reach 0.14% d.b, while 2 cm sample thickness was able to reach the least of 0.19% d.b of EMC until the end of drying process. Furthermore, 1 cm and 2 cm sample took 6 hours and 10 hours, respectively to reach equilibrium state, as compared with 0.5 cm that took three hours. Sample thickness can be related to the distance required for the heat to reach water molecule, as well as the distance required for the moisture to escape through the surface. Therefore, the thicker the sample, the slower the moisture loss, as well as the longer the drying time. The results indicate significant influence of sample thickness towards drying kinetic of sago starch. Similar observation was found for pestil (starch-grape juice mixture) and potato slices by Maskan et al. (2002) and Boutelba et al. (2018). Further explanation on the effect on sample thickness to sago starch drying rate is shown in Figure 4.35 below.

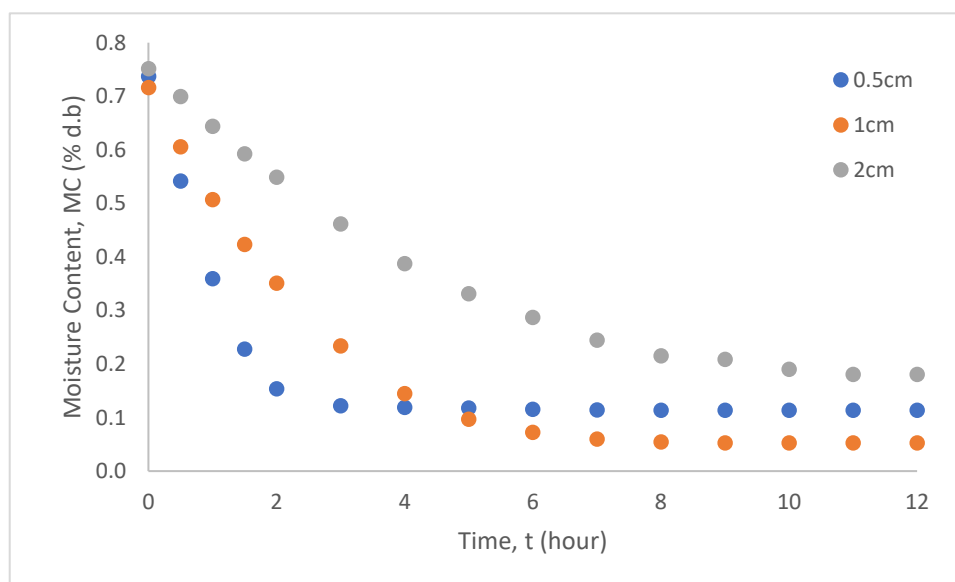


Figure 4.34: Moisture content at different sample thickness

Figure 4.35 shows the drying rate as a function of time at different sample thickness of 0.5, 1 and 2 cm. The results show that 0.5 cm sample has the highest initial drying rate, followed by 1 cm and 2 cm sample thickness. This indicates that thinner sample has less mass that allowed faster heat transfer to the bottom of sample. Meanwhile, thicker samples have more mass that also means more hindrance for heat transfer and moisture loss, thus slowing the drying rate. It also can be seen from the figure that the initial phase drying rate increased as the sample thickness decreased. As a conclusions, this result indicates that sample thickness has significant effect on drying rate of sago starch.

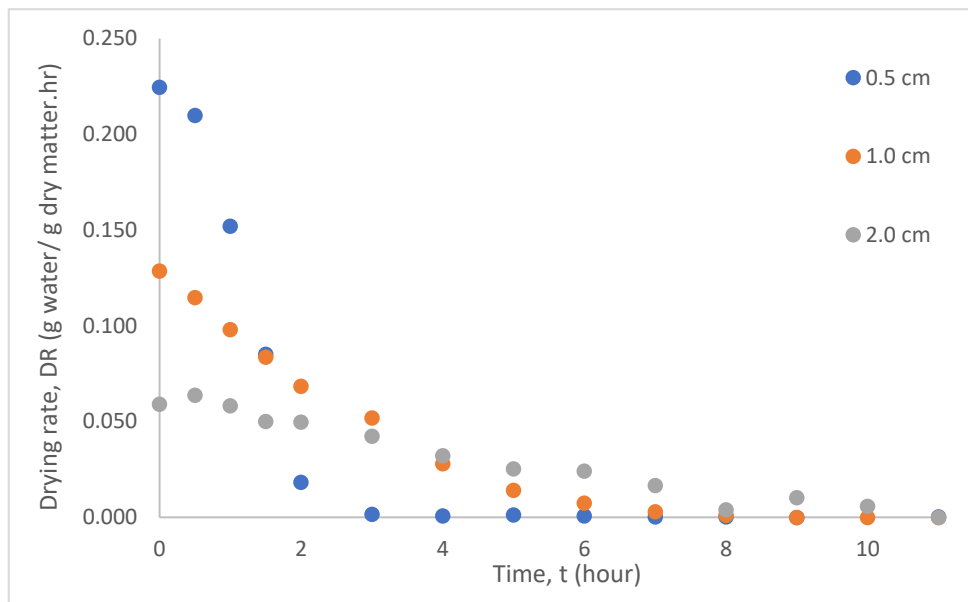


Figure 4.35: Drying rate at different sample thickness

4.5.4 Effect of Sample Position to Sago Starch Drying Kinetic

Figure 4.36 below shows sago starch drying curves of moisture content versus time for different sample positions of A, B and C. Figure 4.36 shows that all samples have a similar curve of moisture content over time during drying process. This indicates that all

samples have similar drying kinetic even at different positions inside oven. Regardless of their position inside oven, all samples probably had received equal amount of heat simultaneously. This indicates that sample position does not have significant effect towards the drying kinetic of sago starch, subjected to the type of drying oven used.

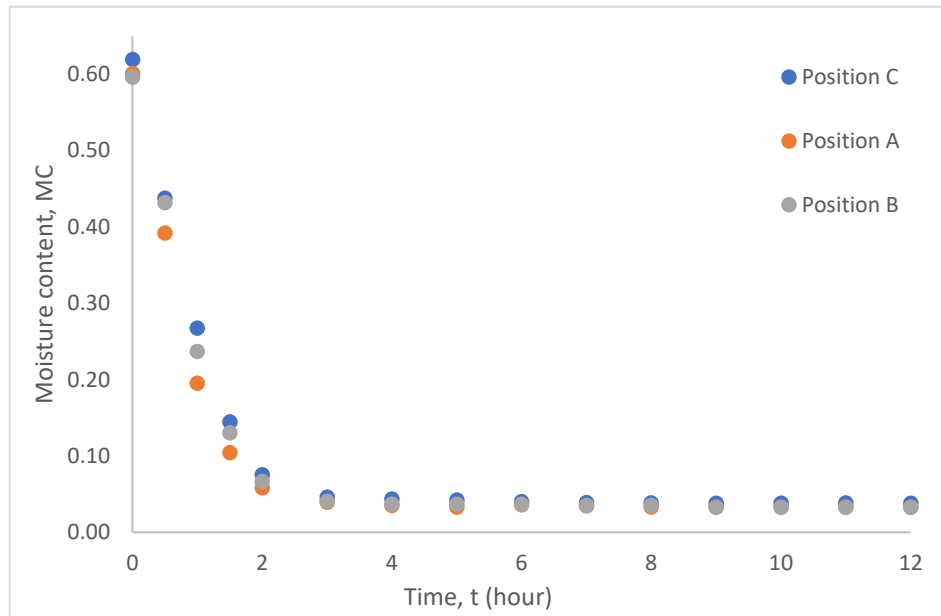


Figure 4.36: Moisture content at different sample position

Further explanation on the effect of sample position to sago starch drying kinetic is shown in Figure 4.37 below. Figure 4.37 shows the changes in drying rate as a function of time at sample position of A, B and C according to Figure 3.6. Sample at position A (lower level-side) has the highest initial drying rate, while sample at position B (lower level-middle) has the lowest initial drying rate probably as the effect of air flow direction that is more rapid at the edge of the oven, as compared to at the middle-opposite fan. Moreover, after one hour, sample at position C (upper level-middle) shows the highest drying rate. It is assumed that the amount of hot air is more at the upper level, as compared to lower level since the hot air

is lighter than cooler air. However, the drying rate difference was not significant; in addition, all three samples have a similar drying rate curve pattern.

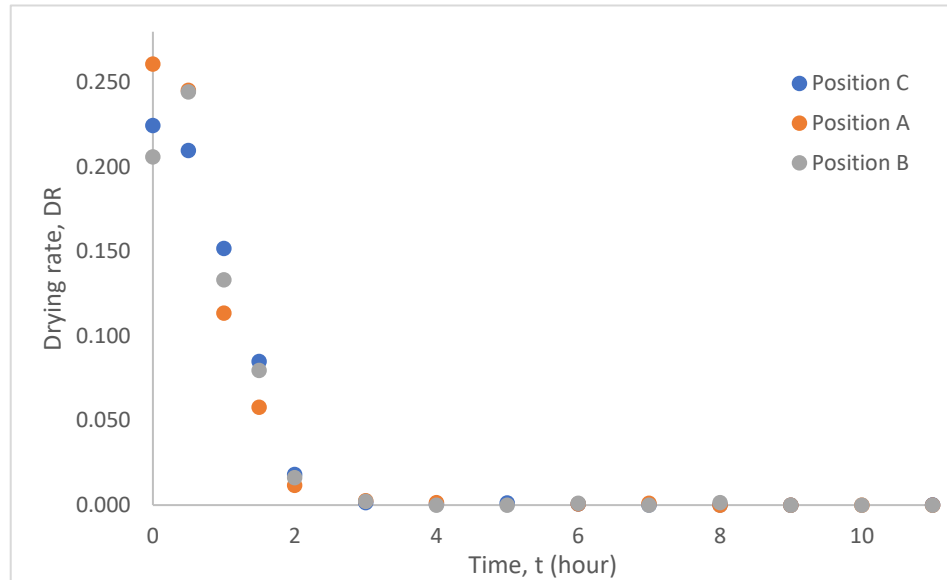


Figure 4.37: Drying rate at different sample position

In conclusion, it is suggested that sago samples can be put anywhere inside the oven without possible difference drying behavior.

4.5.5 Effect of Drying Methods to Sago Starch Drying Kinetic

The effect of drying methods to sago starch drying kinetic is shown in Figure 4.38. Figure 4.38 shows the drying curves of moisture content versus time for different drying methods of continuous drying-natural convection oven (60°C), intermittent drying-natural convection oven (60°C), sun drying, microwave drying and freeze drying.

Figure 4.38 shows that microwave drying resulted in the steepest curve or moisture content reduction over time, followed by intermittent drying, freeze drying, sun drying, and finally continuous drying with natural forced oven. This indicates that microwave drying

allows the fastest drying time followed by intermittent drying, freeze drying, sun drying, and finally continuous drying with natural forced oven. It can be deduced that radiation drying has the most time efficiency, followed by intermittent, freeze, sun, and continuous oven drying. This is because the energy transferred through radiation is probably more powerful than the energy from heat.

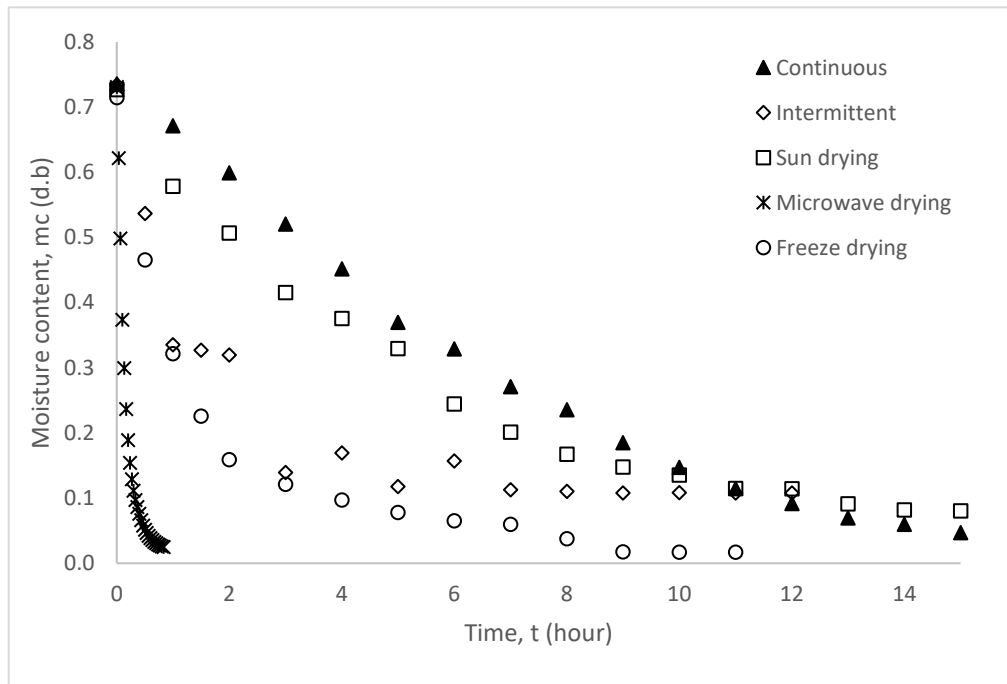


Figure 4.38: Drying curve of sago starch at different drying methods

Figure 4.38 also shows that the drying time required to dry sago starch decreased in intermittent drying as compared with continuous and sun drying. The drying time of sago starch to moisture content of 9% in continuous and sun drying was 9 hour. It is reduced to 4 hour when dried intermittently. This result shows that sago starch can be dried faster through intermittent drying. Intermittent drying is known as a time and energy saving process as it allows moisture in the sample to naturally level themselves during heater-off time. This is because intermittent drying allows heat-off drying phase without extending drying time;

thus, allows energy saving with no effect on productivity. To conclude, regarding overall concern on economic efficiency, intermittent drying seems to be the best option for sago starch drying method.

It also can be seen in Figure 4.38 that continuous drying has almost similar drying kinetic with sun drying. Similar result was obtained by Arslan and Erbakan (2016) when onion slices were dried through oven-continuous and sun drying. This result might be influenced by the wind speed that can enhance drying process. Sun drying can be opted to cut energy cost, but attention should be paid on hygiene problem. Therefore, it can be suggested that intermittent drying is the best way to reduce energy cost and to maintain hygienic condition for sago starch.

Figures 4.38 also depicts that for microwave and freeze drying, drying time required to produce 9% d.b moisture content decreased to 0.8 hour and 3.5 hour, respectively as compared with continuous drying. This result suggests that radiation and freezing-based drying are less time consuming than hot air-convection drying. Arslan and Erbakan (2016) also had found similar results in which microwave drying dried onion slices faster than continuous oven drying. However, microwave drying had caused obvious surface hardening on sago starch sample due to harsh radiation exposure. For sago starch, higher watt may turn the sample surface to dark brown color indicating caramelization of glucose compound in the starch due to over-heating. Meanwhile, freeze drying is also not suitable for starch and powdered material since it has tendency for mass loss due to vacuum suction during the beginning of the drying process.

Drying kinetic of sago starch samples are further studied through its drying rate curve which can be found in Figure 4.39. Figure 4.39 shows the changes in drying rate as a function

of time at different drying processes. All results show that sago starch samples dried at falling rate period with no constant rate period which strongly indicate that sago starch is a type of hygroscopic food. Figure 4.39 shows comparison between continuous, intermittent and sun drying as a group of convection drying. Initial drying rate in continuous drying was lower than sun drying as there was no air flow used in the continuous drying experiment. Intermittent has the highest initial drying as per expected since heat-off time allows moisture to mobilized to the surface that will increase the drying rate during heating time. Both the drying rate of intermittent and sun drying were fluctuating which indicates that sun drying is a type of intermittent drying but occurred naturally. However, the gap between up and down points were not as big as that of intermittent oven-drying. It is probably due to heat and moisture transfer is still happen along the process of sun drying, but there might be sometimes that the quantity is lower than the other times which caused the intermittent effect. Allaf et al. (2014) are among other researchers that also had addressed sun drying as natural intermittent drying.

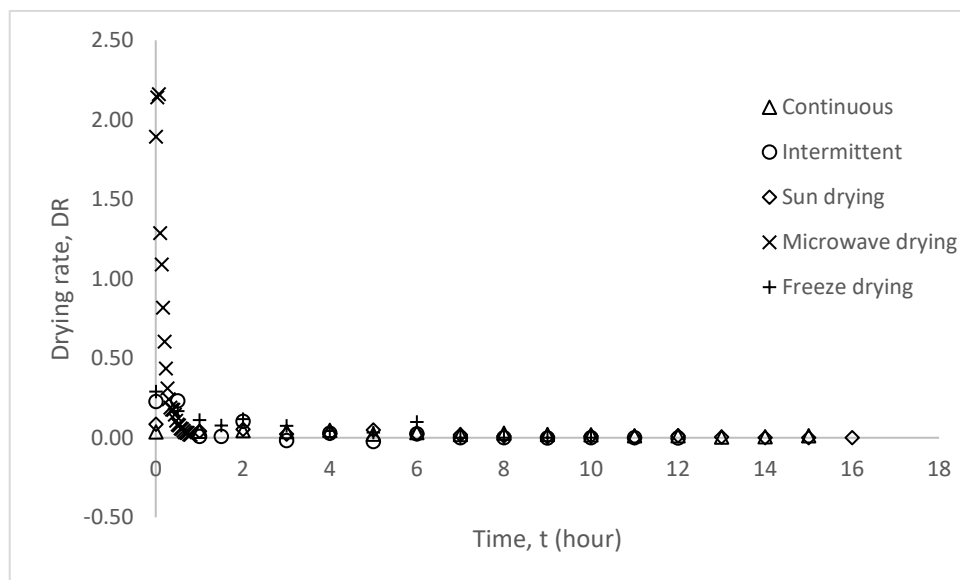


Figure 4.39: Drying rate curve of sago starch at different drying methods

The initial drying rate for microwave drying was the highest among all the drying methods probably due to all the energy from radiation being absorbed by the moisture on the surface only which caused high volume of moisture transfer on the surface. Figure 4.39 also shows the drying rate of microwave drying was increased before it moved to falling rate period. Sago starch samples exhibited a heating up period of up to 3.6 minutes during microwave drying. This shows radiation drying especially in low output power of 200 W such as used in this experiment required few minutes to transfer its heat to the sample. The drying rate decreased gradually after the critical point before the drying process ended within one hour.

It is also depicted that the drying rate over time for freeze drying of sago starch sample. The drying occurred in a falling rate period which indicates the porosity of the sago starch sample in the vacuum tube. The slope was steeper at the beginning of drying until one and half an hour. This stage is known as primary drying period which occurred in forced convection environment and was influenced by the sample thickness as well as material cellular structure (Oyinloye & Yoon, 2020). The later stage is known as secondary drying which normally covers around 30 to 40% of the drying process in freeze drying; however, removes smaller amount of moisture as compared with the primary drying. According to Oyinloye and Yoon (2020), the slower drying rate at this stages might be influenced by the formation of small ice crystal within the sample which can hindered the sublimation process in freeze drying.

4.5.6 Concluding Remarks

The results from this section show that sago starch drying kinetic is affected by drying temperature, air velocity, sample thickness and drying method. Sample thickness does not

give effect to sago starch drying kinetic. This indicates that these variable are important to be noted during drying process.

4.6 Moisture Diffusivities of Sago Starch

4.6.1 Sago Starch Moisture Diffusivities for Different Drying Temperatures

Drying data were subjected to Fick diffusion model and the result of moisture diffusivities (D_{eff}) of sago starch in different temperature as shown in Figure 4.40. In this experiment, it was determined that D_{eff} was highest at 80°C ($1.268 \times 10^{-9} \text{ m}^2/\text{s}$); followed by 70°C ($8.799 \times 10^{-10} \text{ m}^2/\text{s}$), 60°C ($7.462 \times 10^{-10} \text{ m}^2/\text{s}$), and 50°C ($7.086 \times 10^{-10} \text{ m}^2/\text{s}$). Figure 4.40 shows that D_{eff} was significantly affected by drying temperature, in the manner that it increased as the temperature increased. The results also show that the D_{eff} are following the Arrhenius's Law as in Equation 4.10 below.

$$D_{eff} = D_0 \exp \left[\frac{-E_a}{R_g T} \right] \quad \text{Equation 4.10}$$

(Baini & Langrish, 2007)

Where D_0 is the pre-exponential factor of Arrhenius equation (m^2/s), R_g = is the constant of perfect gases (8.3143 kJ/kmol.K) and T is the drying temperature in Calvin.

D_{eff} value is related to the moisture removal speed; thus, higher D_{eff} means more rapid moisture movement and removal, as shown by the high D_{eff} for high temperature. The calculated values were ranged from 7.086×10^{-10} to $1.268 \times 10^{-9} \text{ m}^2/\text{s}$ which are within the general range for food materials that is 10^{-13} to $10^{-6} \text{ m}^2/\text{s}$ (Thao & Noomhorm, 2011).

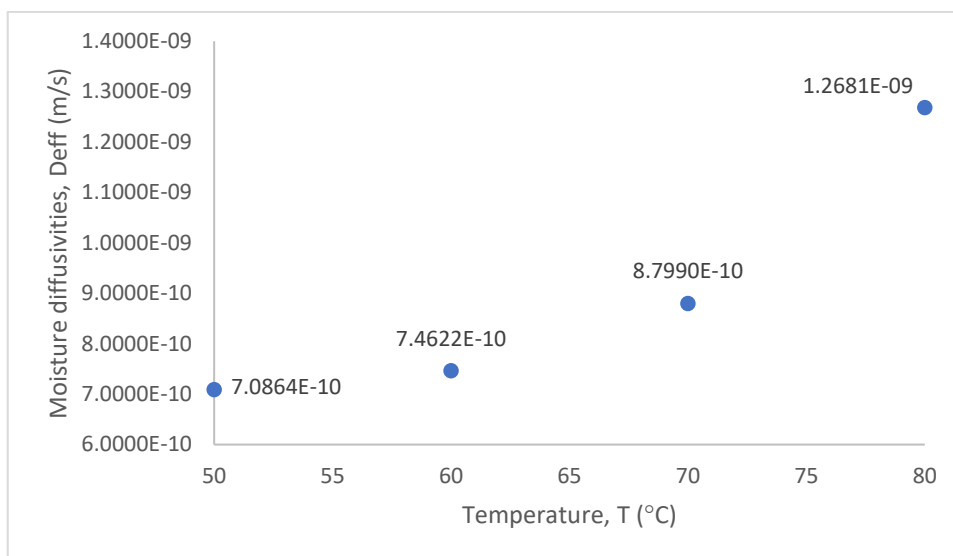


Figure 4.40: Moisture diffusivities at different drying temperature

4.6.2 Sago Starch Moisture Diffusivities for Different Air Velocity

Drying data were subjected to Fick diffusion model and the result of moisture diffusivities (D_{eff}) of sago starch in different temperature is shown in Figure 4.41. In this experiment, the highest moisture diffusivities were at 0.965 m/s ($5.773 \times 10^{-9} \text{ m}^2/\text{s}$); followed by 0.676 m/s ($5.592 \times 10^{-9} \text{ m}^2/\text{s}$), 60.386 m/s ($2.899 \times 10^{-9} \text{ m}^2/\text{s}$), and 0 m/s ($2.143 \times 10^{-9} \text{ m}^2/\text{s}$). The result shows that moisture diffusivities were significantly affected by air velocity with $p < 0.05$. This indicated that the introduction of air flow in the oven accelerated the moisture removal speed of sago starch which increased as the velocity increased. The calculated values were ranged from 2.143×10^{-9} to $5.773 \times 10^{-9} \text{ m}^2/\text{s}$ which are within the general range for food materials that is 10^{-13} to $10^{-6} \text{ m}^2/\text{s}$ (Thao & Noomhorm, 2011)

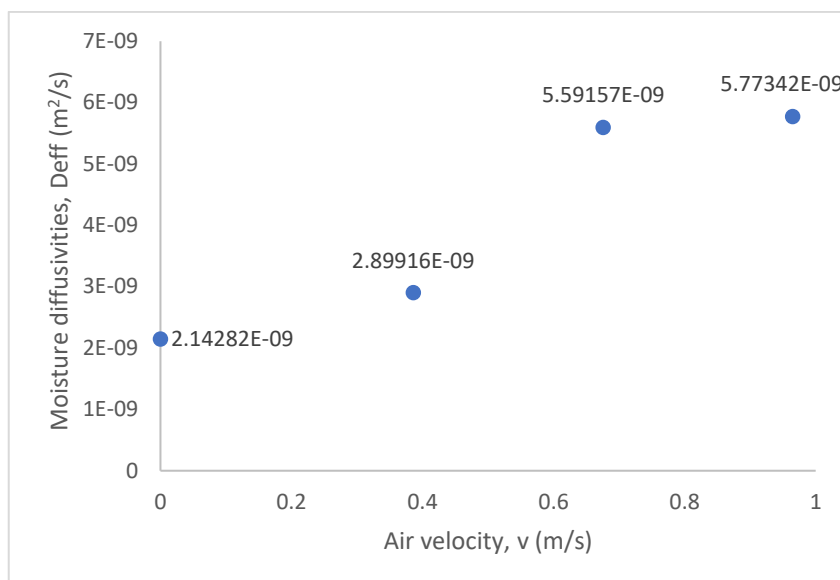


Figure 4.41: Diffusion coefficient at different air velocity

4.6.3 Sago Starch Moisture Diffusivities for different Sample Thickness

The results from drying profile above show that drying had occurred at falling rate period. Therefore, Fick diffusion model can be applied to the drying data to obtain moisture diffusivities (D_{eff}) of sago starch in different thickness as shown in Figure 4.42. In this experiment, it was determined that D_{eff} was the highest at 2 cm thickness ($8.2 \times 10^{-10} m^2/s$); followed by 1 cm ($1.4 \times 10^{-9} m^2/s$) and 0.5 cm ($2.5 \times 10^{-9} m^2/s$). Figure 4.42 clearly shows that D_{eff} was significantly affected by sample thickness, in the manner that it decreased as the thickness increased. As D_{eff} value represents the moisture removal speed based on internal resistance, low D_{eff} in 2 cm sample indicates that the thicker the sample the internal resistance becomes more intensive. Similar with results in two section above, the calculated values were ranged from 8.2×10^{-10} to $2.5 \times 10^{-9} m^2/s$ which are within the general range for food materials that is 10^{-13} to $10^{-6} m^2/s$ (Thao & Noomhorm, 2011).

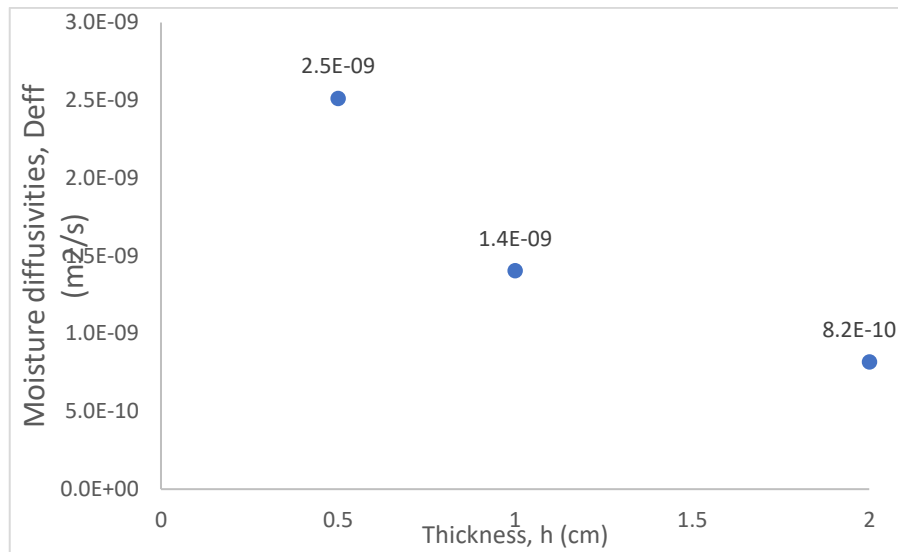


Figure 4.42: Moisture diffusivities at different sample thickness

4.6.4 Sago Starch Moisture Diffusivities for different Drying Methods

The results from drying profile above shows that drying had occurred at falling rate period. Therefore, Fick diffusion model can be applied to all the drying data to obtain effective moisture diffusivities (D_{eff}) of sago starch in different thickness as shown in Figure 4.43.

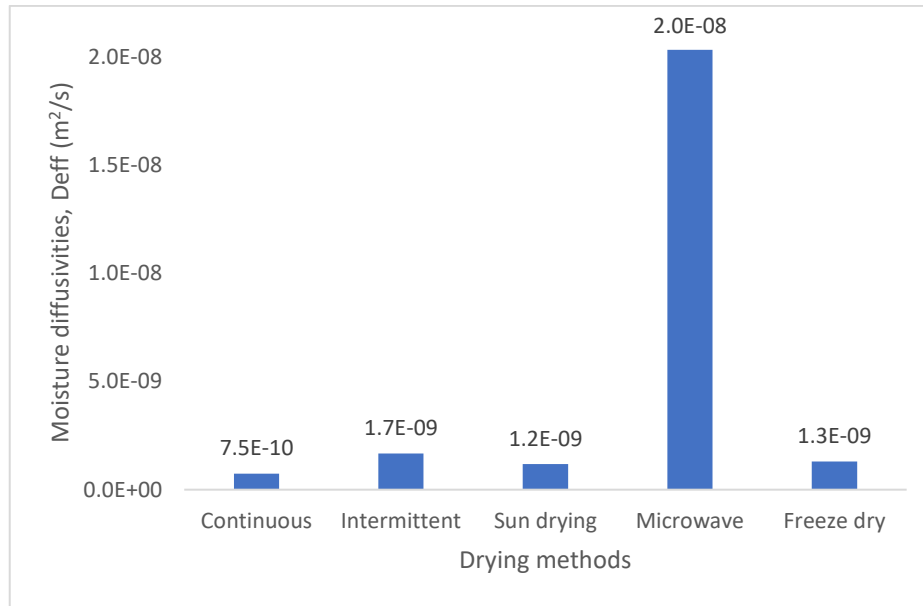


Figure 4.43: Moisture diffusivities in different drying methods

In this experiment, it was determined that microwave drying method resulted in the highest at effective moisture diffusivities of $2.0 \times 10^{-8} m^2/s$; followed by intermittent drying at $1.7 \times 10^{-9} m^2/s$, freeze drying ($1.3 \times 10^{-9} m^2/s$), sun drying ($1.2 \times 10^{-9} m^2/s$), and continuous drying at $7.5 \times 10^{-10} m^2/s$. It is noted that the moisture diffusivities results have similar trend with EMC results, but only for heat convection drying type of methods that are continuous, intermittent and sun drying. The results indicate that the microwave radiation increased the moisture movement significantly. However, this high-speed moisture removal that occurred on the surface and within the sample had also caused significant surface hardened to the extent of color change due to caramelization. The De_{eff} result for microwave drying obtained from this experiment was in the same range with the De_{eff} obtained by Arslan and Erbakan (2016) through onion drying at 210 watt ($1.254 \times 10^{-9} m^2/s$); however it was slightly faster than the result of yam drying ($4.44 \times 10^{-9} m^2/s$) obtained by Murthy (2014).

The result of freeze drying D_{eff} obtained from this study was faster than the results of D_{eff} for guava and papaya obtained by Hawlader et al. (2006) which was in the range between 6 to $10 \times 10^{-10} \text{ m}^2/\text{s}$. This result might be influenced by the difference of molecular structure between sago starch and guava/papaya fruit, in which the former is in powder form and the latter is in fruit-slice form. Moisture travels easier in powdery material such as starch and milk powder as compared with compact-solid material such as fruit or wood.

The moisture diffusivities for intermittent drying at 60°C was higher than that of continuous at 60°C and sun drying. This indicates that when sago starch is drying at same temperature and condition, an introduction of intermittent drying will cause the changes in moisture removal behavior in the manner of increasing movement speed. This effect can be explained by the moisture leveling phenomenon that occurred within the sample during the heat-off period. The result obtained is in agreement with the result obtained by Baini and Langrish (2007) for drying of banana in which intermittent drying ($1.58 \times 10^{-9} \text{ m}^2/\text{s}$) has higher D_{eff} as compared with continuous drying ($1.23 \times 10^{-9} \text{ m}^2/\text{s}$).

Sun drying has higher D_{eff} value than continuous drying which indicates that the effect of wind as air flow, as well as natural intermittent concept applied in the methods had increased the moisture diffusion rate. However, when this result is compared with the results from drying at different temperature above (section 4.6.1), the D_{eff} for continuous drying at 70°C was $8.80 \times 10^{-10} \text{ m}^2/\text{s}$, and at 80°C was $1.27 \times 10^{-9} \text{ m}^2/\text{s}$. Both results were higher than that of sun drying ($8.52 \times 10^{-10} \text{ m}^2/\text{s}$). This indicates that drying temperature has more influence than drying methods, in terms of the comparison between continuous-oven and sun drying. This results is in agreement with the results obtained by Arslan and Erbakan(2016) during the drying of onion slices. Sun drying method also has lower D_{eff}

value than intermittent drying method which indicates that a proper time-off period play an important role in determining drying effect.

4.6.5 Concluding Remarks

The results from this section show that sago starch moisture diffusivities were affected by drying temperature, air velocity, sample thickness and drying methods. This indicates that these variables are useful to increase drying efficiency.

4.7 Assessment of Drying Model for Sago Starch

Several drying models have been used to assess the drying kinetics for sago starch, and the description of the models is given in Table 2.2, Section 2.6.1, Chapter 2, page 41. The analysis includes the applicability of the models in predicting the drying profiles for sago starch for different temperatures.

4.7.1 Assessment of Empirical Model for Sago Starch Drying Kinetic

4.7.1.1 Effect of Drying Temperature to Sago Starch`s Empirical Model Assessment

Ten thin layer drying models were fitted to the experimental data of moisture ratios at different temperatures through non-linear regression by using solver in Excel. All these models have been found to be well-fitted experimental moisture content for materials that were dried continuously. The non-linear regression results are shown in Table 4.8 which consists of drying constants, standard error (SE) and correlation coefficient (R^2).

Table 4.8: Results of fitting model criteria; drying constants, Standard Error (SE) and correlation coefficient (R^2) from different sago starch drying temperature

Model	Drying temperature			
	50 °C	60 °C	70 °C	80 °C
Lewis Newton	SE= 0.055	SE= 0.042	SE= 0.047	SE= 0.035
	k= 0.147	k= 0.166	k= 0.231	k= 0.399
	R^2 =0.998	R^2 =0.999	R^2 =0.999	R^2 =0.998
Page	SE= 0.020	SE= 0.013	SE= 0.016	SE= 0.006
	k= 0.043	k= 0.076	k= 0.094	k= 0.225
	n= 1.607	n= 1.405	n= 1.537	n= 1.515
	R^2 = 0.996	R^2 = 0.998	R^2 = 0.998	R^2 = 1.000
Henderson and Pabis	SE= 0.048	SE= 0.036	SE= 0.041	SE= 0.032
	k= 0.163	k= 0.180	k= 0.251	k= 0.420
	a= 1.116	a= 1.086	a= 1.103	a= 1.065
	R^2 =0.979	R^2 =0.989	R^2 =0.987	R^2 =0.991
Modified Henderson and Pabis	SE= 0.055	SE= 0.041	SE= 0.047	SE= 0.151
	k= 64.793	k= 20.994	k= 36.668	k= 14.008
	a= 1.552	a= 3.328	a= 2.154	a= 2.503
	c= -1.550	c= 3.328	c= -2.155	c= -2.503
	g= 0.147	g= 0.167	g= 0.231	g= 0.399
	h= 11.019	h= 3.957	h= 13.268	h= 13.760
	R^2 =0.982	R^2 =0.990	R^2 =0.999	R^2 =0.991
Wang and Singh	SE= 0.015	SE= 0.009	SE= 0.018	SE= 0.067

	a= -0.099	a= -0.120	a=-0.163	a= -0.217
	b= 0.002	b= 0.004	b= 0.006	b= 0.011
	R ² = 0.998	R ² = 0.999	R ² = 0.998	R ² = 0.965
Logarithmic	SE= 0.193	SE= 0.198	SE= 0.192	SE= 0.164
	k= 62.884	k= 21.596	k= 41.879	k= 12.830
	a= 0.627	a= 0.665	a= 0.761	a= 0.871
	c= 0.373	c= 0.335	c= 0.239	c= 0.129
	R ² =0.455	R ² =0.499	R ² =0.545	R ² =0.692
Two term	SE= 0.305	SE= 0.304	SE= 0.248	SE= 0.188
	k= 62.884	k= 21.596	k= 41.879	k= 12.830
	a= 3.344	a= 0.934	a= 2.975	a= 2.699
	c= -2.344	c= 1.934	c= -1.975	c= -1.699
	g= 14.883	g= 31.195	g= 16.979	g= 13.599
	R ² =0.455	R ² =0.499	R ² =0.545	R ² =0.692
Approximation of Diffusion	SE= 0.026	SE= 0.016	SE= 0.019	SE= 0.009
	k= 0.294	k= 0.342	k= 0.446	k= 0.766
	a= 9.257	a= 7.621	a= 9.433	a= 9.116
	g= 0.930	g= 0.899	g= 1.118	g= 1.121
	R ² = 0.994	R ² = 0.998	R ² = 0.997	R ² = 0.999
Midili and Kucuk	SE= 0.281	SE= 0.283	SE= 0.241	SE= 0.187
	k= 62.884	k= 21.596	k= 41.879	k= 12.830
	a= 1.000	a= 1.000	a= 1.000	a= 1.000
	b= 0.020	b= 0.018	b= 0.009	b= 0.004
	R ² =0.080	R ² =0.187	R ² =0.408	R ² =0.656

Verma et al.	SE= 0.026	SE= 0.018	SE= 0.019	SE= 0.009
	k= 0.294	k= 0.342	k= 0.446	k= 0.766
	a= 9.308	a= 7.662	a= 9.506	a= 8.771
	g= 0.330	g= 0.307	g= 0.499	g= 0.863
	R ² = 0.994	R ² = 0.998	R ² = 0.997	R ² = 0.999

The suitability of the model was chosen based on low standard error and high correlation coefficient value. Standard error (SE) was used to determine the average difference between the experimental values and the predicted data. The result from Table 4.8 shows that low values of standard errors (SE<0.1) and high value of correlation coefficient (R²) were found for all models except for three models namely Logarithmic, Two Term and Midili & Kucuk model. The R² values also were considerably high (R² >0.900) which indicated that the seven models were applicable for describing the drying behaviour of sago starch at drying temperature of 50, 60, 70 and 80°C.

To confirm the relationship, an exponential curve was generated between experimental and predicted data for each temperature by using Lewis model as a representative model. The results indicate good agreements between the predicted moisture content from Lewis model and experimental values for all drying temperatures as shown in Figure 4.44.

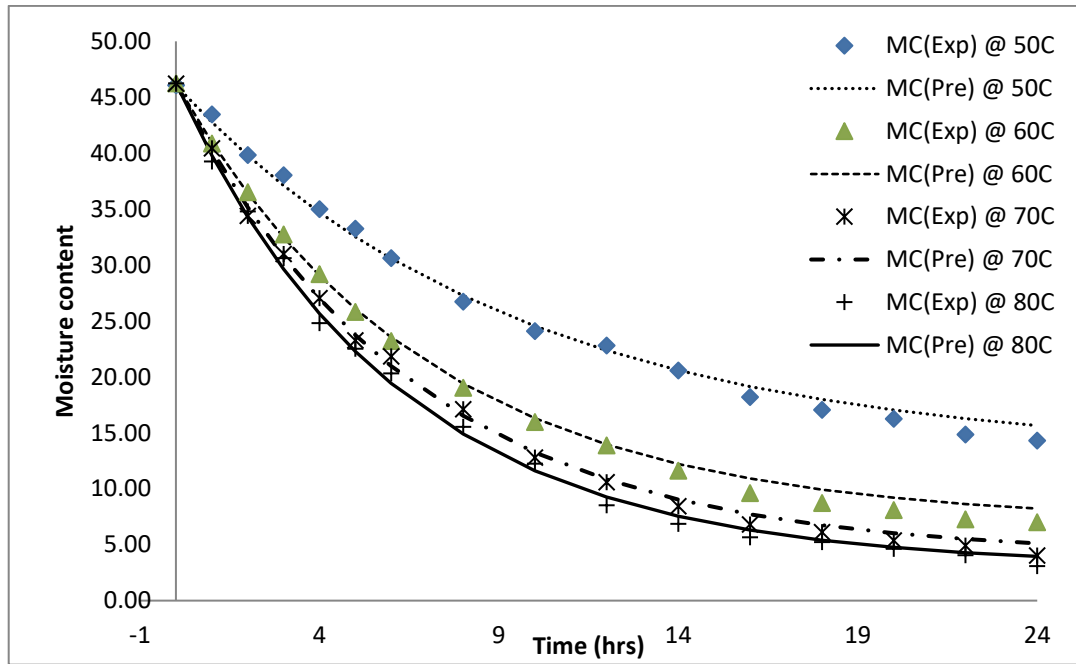


Figure 4.44: Predicted and experimental moisture content of sago starch using Lewis model for 50, 60, 70 and 80°C

It can be seen from Figure 4.44 that the agreement between both experimental and predicted data are excellent which means they are highly identical with each other. The correlation coefficient (R^2) values from Table 4.8 were also considerably high ($R^2 > 0.98$) which strongly suggest that these models are applicable for describing the drying behaviour of sago starch at drying temperature of 50, 60, 70 and 80°C. The results of correlation coefficient can be presented by generating a linear graph of experimental and predicted data for each temperature as shown in Figure 4.45

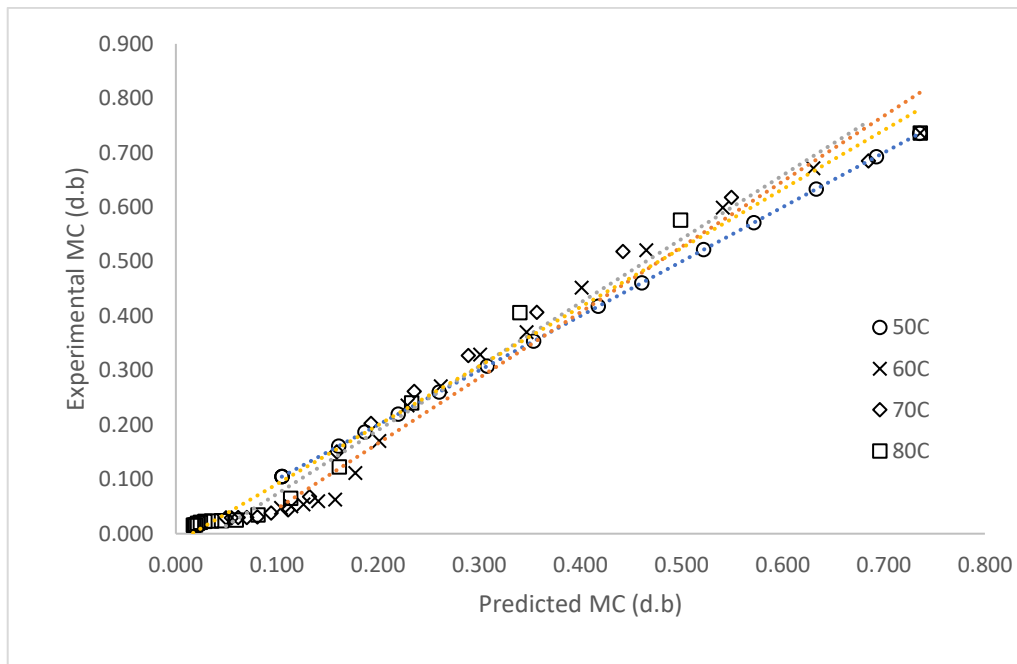


Figure 4.42: Comparison of experimental and predicted value of Lewis model

Figure 4.45 show that the generated trendline was linear from origin. This behavior indicates that the predicted model is a good fit for the actual drying data. Therefore, it is suggested that the Lewis model, as well as another six models can be used for the drying process prediction of sago starch.

4.7.1.2 Effect of Drying Method to Sago Starch`s Empirical Model Assessment

In this section, the suitability of ten drying models that are commonly used to predict the drying kinetic of various fruits were observed. These models were fitted to the experimental data of moisture ratios at different sample thickness through non-linear regression by using solver in Excel. The result is shown in Table 4.9 which consists of standard error (SE), drying constant and correlation coefficient (R^2). For continuous oven-drying, the models that can be used for describing the thin layer drying of sago starch have been discussed above.

Table 4.9: Results of fitting model criteria; drying constants, standard error (SE) and correlation coefficient (R^2) from different sago starch drying method

Model	Methods				
	Continuous	Intermittent	Sun drying	Microwave	Freeze drying
Lewis Newton	SE= 0.042	SE= 0.029	SE= 0.020	SE= 0.011	SE= 0.360
	k= 0.166	k= 0.742	k= 0.227	k= 6.901	k= 0.098
	R^2 =0.999	R^2 = 0.986	R^2 = 0.996	R^2 = 0.999	R^2 = 0.948
Page	SE= 0.013	SE= 0.027	SE= 0.017	SE= 0.009	SE= 0.097
	k= 0.076	k= 0.786	k= 0.186	k= 7.683	k= 0.350
	n= 1.405	n= 0.854	n= 1.117	n= 1.059	n= 1.024
	R^2 = 0.998	R^2 = 0.988	R^2 = 0.997	R^2 = 0.999	R^2 = 0.946
Henderson and Pabis	SE= 0.036	SE= 0.029	SE= 0.020	SE= 0.009	SE= 0.11
	k= 0.180	k= 0.721	k=0.230	k= 7.083	k= 0.355
	a= 1.086	a= 0.977	a= 1.012	a= 1.027	a= 0.987
	R^2 =0.989	R^2 = 0.986	R^2 = 0.996	R^2 = 0.999	R^2 = 0.947
Modified Henderson and Pabis	SE= 0.041	SE= 0.028	SE= 0.020	SE= 0.003	SE= 0.091
	k= 20.994	k= 0.643	k= 49.938	k= 5.741	k= 2.341
	a= 3.328	a= 0.116	a= 3.801	a= 0.662	a= -0.165
	c= 3.328	c= 0.116	c= -3.801	c= -0.662	c= 0.165
	g= 0.167	g= 0.643	g= 0.227	g= 18.117	g= 0.354
	h= 3.957	h= 10.983	h= 11.019	h= 30.031	h= 12.141
	R^2 =0.990	R^2 = 0.987	R^2 = 0.996	R^2 = 0.999	R^2 = 0.948

Wang Singh	and	SE= 0.009	SE= 0.102	SE= 0.028	SE= 0.069	SE= 0.146
		a= -0.120	a= 0.293	a=0.153	a= 3.848	a= -0.240
		b= 0.004	b= 0.019	b= 0.006	b= 3.472	b= 0.014
		R ² = 0.999	R ² =0.915	R ² =0.994	R ² =0.998	R ² =0.998
Logarithmic		SE= 0.198	SE= 0.029	SE= 0.157	SE= 0.009	SE= 0.159
		k= 21.596	k= 0.747	k= 62.718	k= 7.198	k= -0.003
		a= 0.665	a= 0.970	a= 0.783	a= 1.025	a= -29.983
		c= 0.335	c= 0.011	c= 0.217	c= 0.005	c= -30.716
		R ² =0.499	R ² =0.986	R ² = 0.606	R ² = 0.999	R ² = 0.781
Two term		SE= 0.304	SE= 0.027	SE= 0.210	SE= 0.009	SE= 0.114
		k= 21.596	k= 2.232	k= 62.718	k= 7.079	k= 0.535
		a= 0.934	a= 0.265	a= 2.610	a= 3.704	a= -2.309
		c= 1.934	c= 0.740	c= -1.610	c= -2.677	c= 3.273
		g= 31.195	g= 0.561	g= 19.246	g= 7.077	g= 0.465
		R ² =0.499	R ² = 0.988	R ² = 0.606	R ² = 0.999	R ² = 0.936
Approximation of Diffusion		SE= 0.016	SE= 0.027	SE= 0.016	SE= 0.007	SE= 0.114
		k= 0.342	k= 2.207	k= 0.329	k= 7.394	k= 0.449
		a= 7.621	a= 0.259	a= 8.852	a= 1.072	a= 3.471
		g= 0.899	g= 0.255	g= 1.056	g= 29.732	g= 1.102
		R ² = 0.998	R ² =0.988	R ² =0.997	R ² =0.999	R ² =0.944
Midili Kucuk	and	SE= 0.283	SE= 0.029	SE= 0.203	SE= 0.009	SE= 0.132
		k= 21.596	k= 0.728	k= 62.718	k= 7.142	k= -0.114
		a= 1.000	a= 0.978	a= 1.000	a= 1.029	a= 0.884

	b= 0.018	b= 0.0008	b= 0.008	b= 0.006	b= -0.277
	R ² =0.187	R ² = 0.986	R ² =0.464	R ² =0.999	R ² =0.885
	SE= 0.018	SE= 0.043	SE= 0.016	SE= 0.012	SE= 0.114
	k= 0.342	k= 1.000	k= 0.329	k= 6.310	k= 0.448
Verma et al.	a= 7.662	a= 0.786	a= 8.923	a= 7.851	a= 3.335
	g= 0.307	g= 0.854	g= 0.347	g= 6.230	g= 0.497
	R ² = 0.998	R ² =0.980	R ² =0.997	R ² =0.999	R ² =0.944

Table 4.9 depicts that of all four drying methods, only microwave drying has relatively lower standard error and high correlation coefficient for all fitted models which indicates their reliability for data prediction. By means, all ten models can be used to predict drying kinetic of sago starch dried in the microwave. Figures 4.46 and 4.47 below show the conformation between experimental and predicted moisture content of Midili and Kucuk model as a representative model to show good data fitting for microwave drying of sago starch.

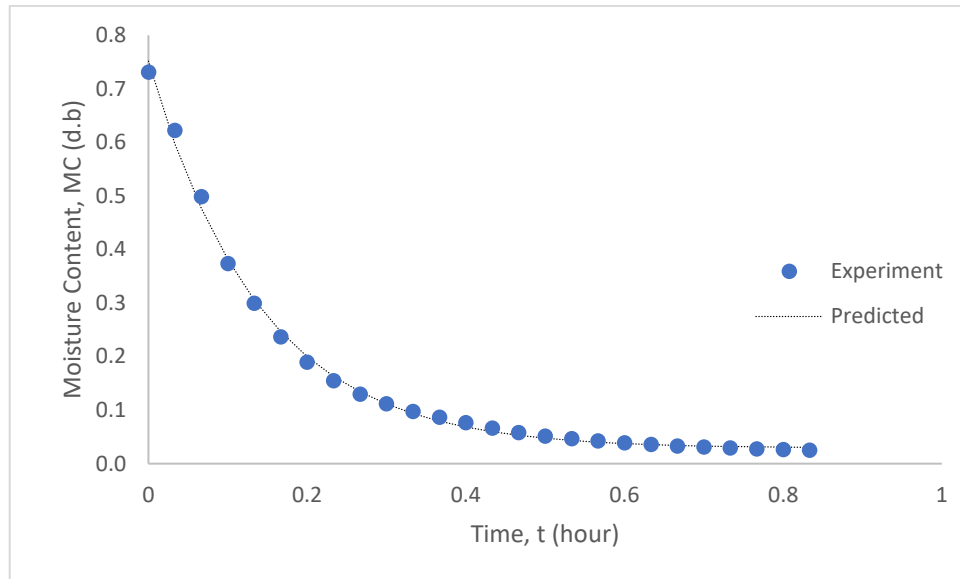


Figure 4.46: Predicted and experimental moisture content of sago starch using Midili & Kucuk model for microwave drying

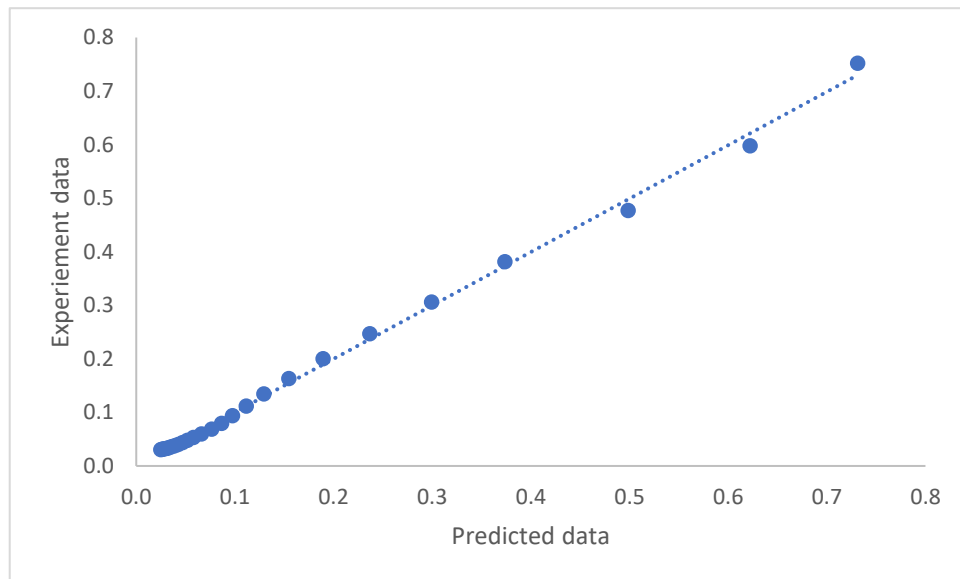


Figure 4.47: Comparison of experiment and predicted value of Midili & Kucuk model for microwave drying of sago starch

According to Kucuk et al. (2014), all listed microwave drying experiment found in their literature had suggested Midili and Kucuk model that is a type of semi-empirical model,

as the most suitable model to predict microwave drying in various foods such as grapes, leek, celery leaves and spinach. However, when microwave combined with others drying methods or tools such as oven, vacuum or fan, more models were successfully regressed such as Page and Wang-Sing model.

The results of model fitting for continuous drying are similar with the results in previous section of 4.7.1.2 which concluded that all models except for Logarithmic, Two-Term and Midili Kucuk are suitable for drying kinetic prediction of sago starch. For intermittent drying, all models generated relatively low standard error during regression analysis, except for Wang and Singh model that has relatively high standard error, as well as low correlation coefficient. Further conformation between experimental and predicted moisture content of Page model as a representative model in intermittent drying as compared with continuous drying is observed as in Figure 4.48 and Figure 4.49.

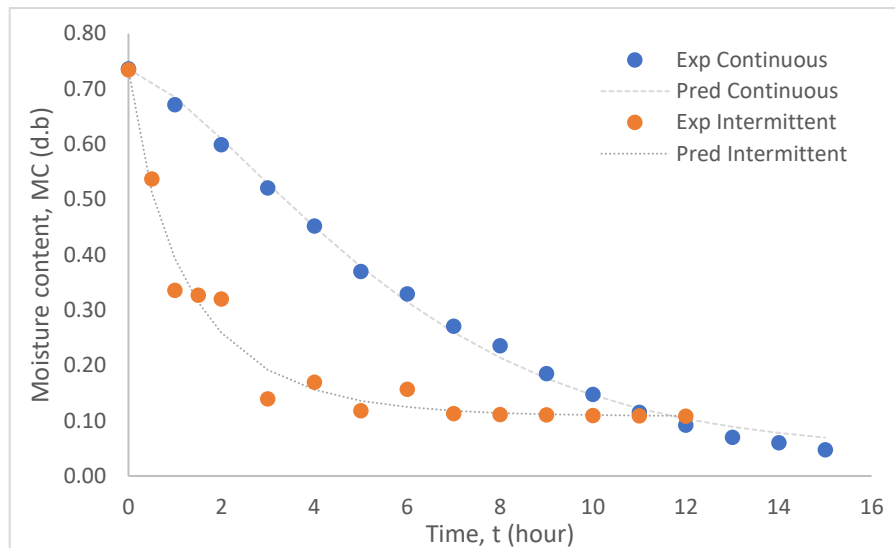


Figure 4.48: Predicted and experimental moisture content of sago starch using Page model for intermittent and continuous drying at 60 °C

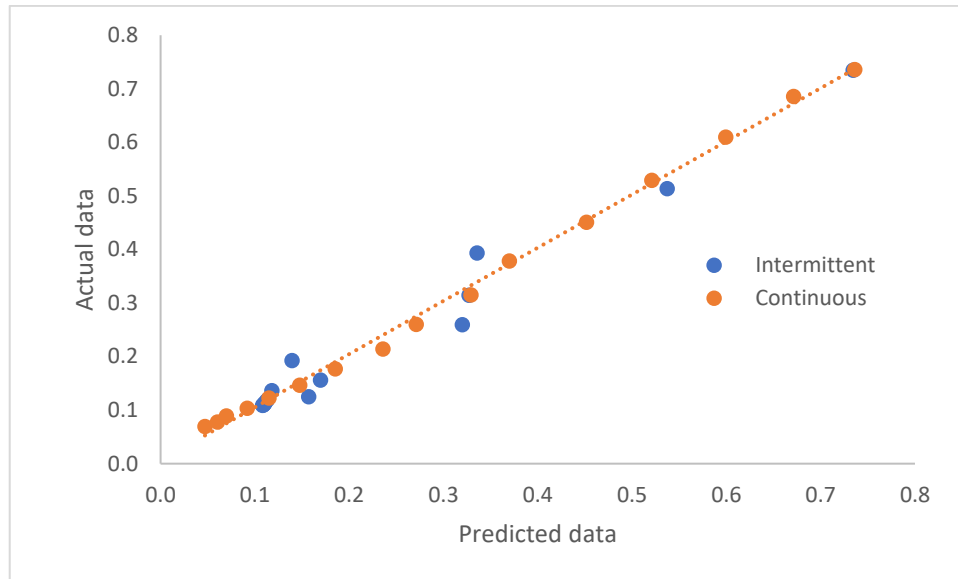


Figure 4.49: Comparison of actual and predicted value of Page model for intermittent and continuous drying of sago starch at 60°C

As for sun drying, the regression results provided low standard error in all models except for in Logarithmic, Two term and Midili & Kucuk model. This results are similar to the results obtained from data fitting of continuous drying at different temperature. Further conformation between experimental and predicted moisture content of Verma model as a representative model in sun drying is observed in Figure 4.50 and Figure 4.51 below.

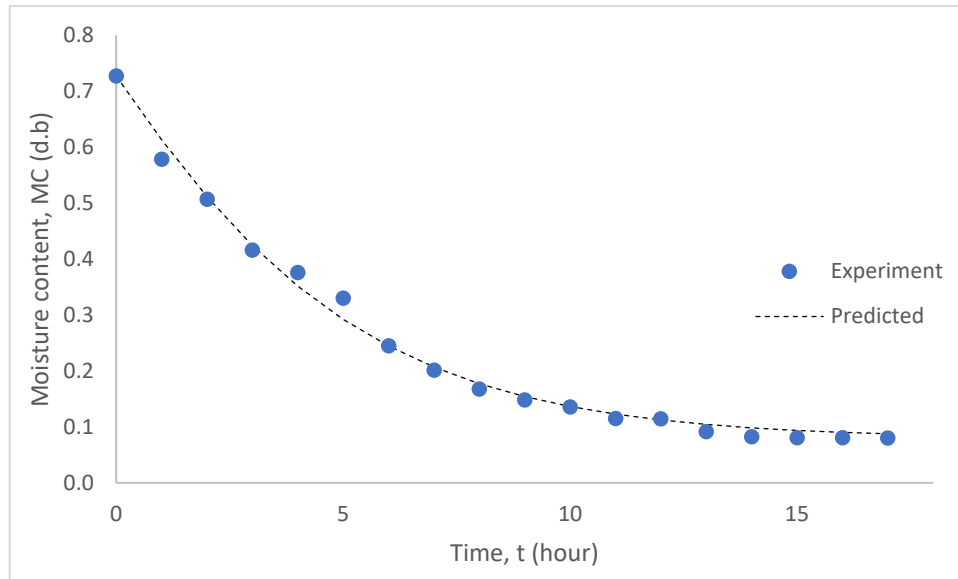


Figure 4.50: Predicted and experimental moisture content of sago starch using Verma model for sun drying

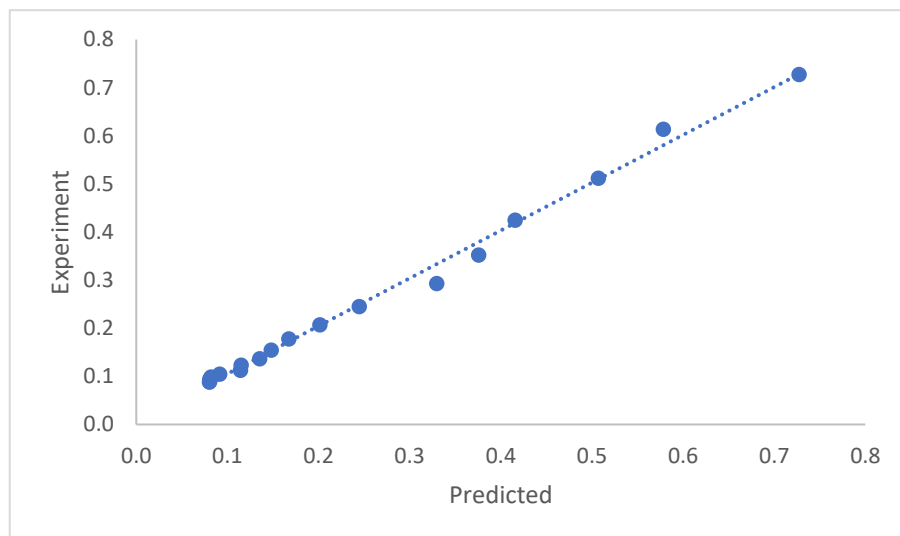


Figure 4.51: Comparison of actual and predicted value of Verma model for sun drying

The results from regression analysis of freeze-drying experiment have showed that all models have high standard error as well as low correlation coefficient. Moreover, the values are significantly high for Midili & Kucuk and Logarithmic model. When the data

points are generated together in a graph of moisture content versus drying time, the results show fair agreement between experimental and predicted data as shown in Figure 4.52 and 4.53 below.

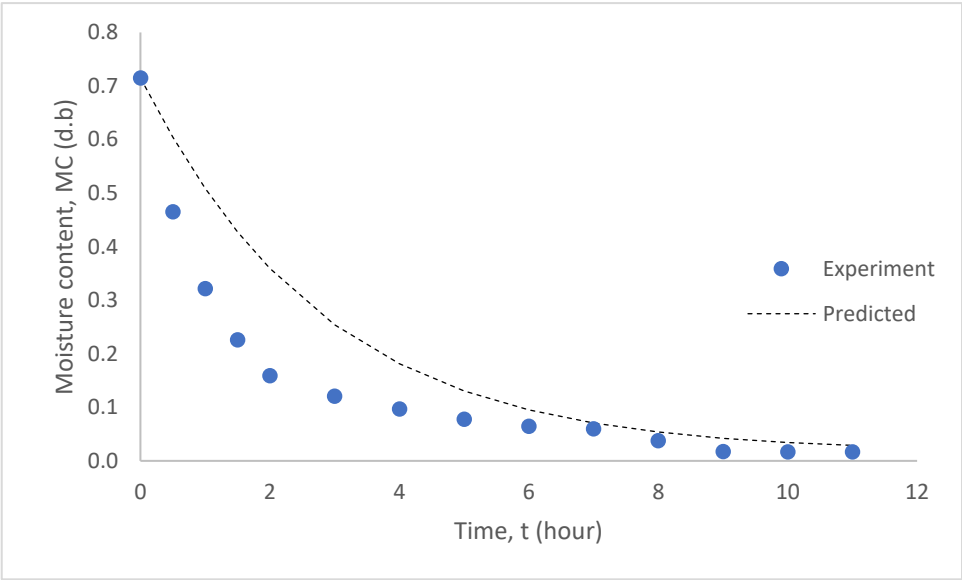


Figure 4.52: Predicted and experimental moisture content of sago starch using Page model for freeze drying

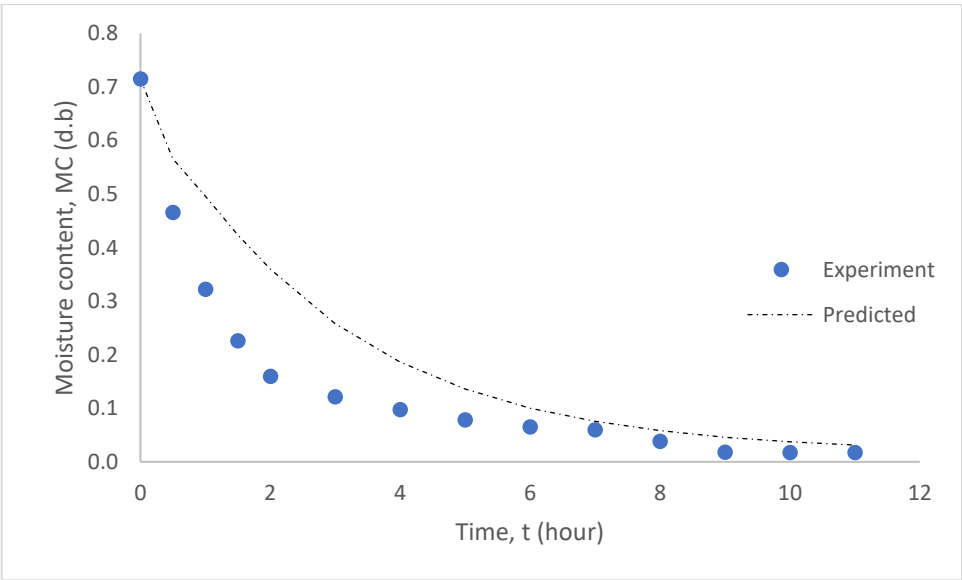


Figure 4.53: Predicted and experimental moisture content of sago starch using Modified Henderson & Pabis model for freeze drying

Figures 4.52 and Figure 4.53 show that most of the data are not matching with the other at the same time. Therefore, it can be deduced that none of the thin layer drying model can be used to predict freeze drying data for sago starch. Furthermore, Figure 4.54 below shows that actual and predicted data from both models did not fall into a straight line which indicate fair agreement between the models and drying data.

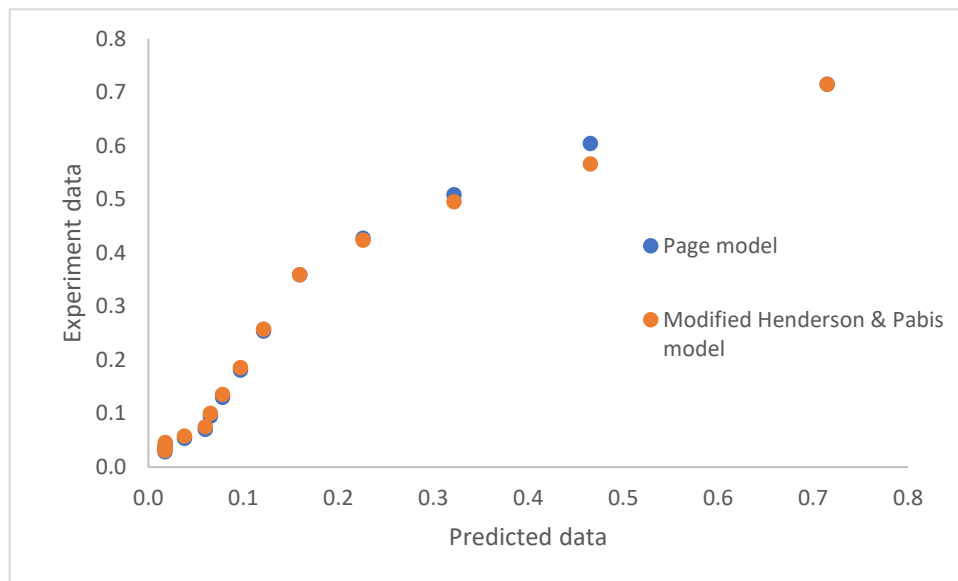


Figure 4.54: Comparison of actual and predicted value of Page and Modified Henderson & Pabis model for freeze drying

4.7.2 Assessment of Simplified Fick Diffusion Model for Sago Starch Drying Kinetic

The activation energy for drying process of sago starch was found to be 17.943 kJ/mol (correlation coefficient, $r = 0.8619$) which fall in the range of E_a values for food materials as stated by Maskan et al, (2002), which is 15 to 40 kJ/mol. In addition, the activation energy for drying temperature of 50 °C, 60 °C, 70 °C, and 80 °C are 17.7 kJ/mol, 18.3 kJ/mol, 18.8 kJ/mol, and 19.4 kJ/mol, respectively. This shows that the activation energy increases as the drying temperature increases.

Furthermore, the applicability of Fickian drying models to describe the drying kinetics of sago starch is analyzed. Standard error (SE) was used to determine the average difference that the experimental values fall from the regression line of Fickian model, while the correlation coefficient (R^2) is used to quantitatively evaluate the model fitting as shown in Table 4.10 below.

Table 4.10: Standard error (SE), correlation coefficient and drying constant results

Drying temperatures (°C)	Standard error (SE)	Correlation coefficient (R^2)	Drying constant (A;B)
50	0.047	0.979	1.112; -0.163
60	0.05	0.983	1.100; -0.190
70	0.04	0.987	1.103; -0.251
80	0.03	0.991	1.065; -0.420

The result from Table 4.10 shows that low values of standard errors ($SE < 0.1$) were found for Fickian model. All the R^2 values were considerably high ($R^2 > 0.9$) which indicated that this model was applicable for describing the drying behavior of sago starch at drying temperature of 50, 60, 70 and 80°C. To compare, the standard error for simplified Fickian model fitting in continuous oven drying at 60 °C was 0.05, while the standard error for empirical model fitting was 0.009. It can be deduced that the standard error of model fitting

can be reduced up to 82% by using empirical model, as compared to simplified Fickian. However, Baini and Langrish (2007) had found a contra results by using the original Fick Diffusion model.

To confirm the relationship, an exponential curve between experimental and predicted data was generated as in Figure 4.55. The results showed that there were good agreements between the predicted moisture content from Fickian model and experimental values for all drying temperatures.

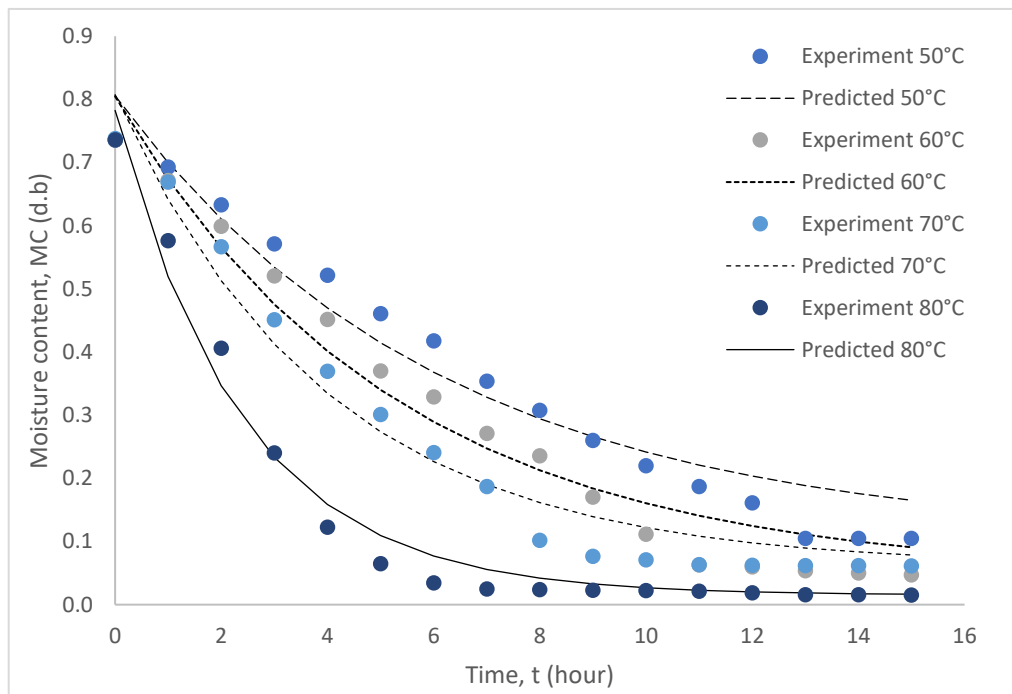


Figure 4.55: Predicted and experimental moisture content of sago starch using Simplified Fickian model for 50, 60, 70 and 80 °C

Furthermore, the results of correlation coefficient are presented by generating a linear graph of experimental and predicted data for each temperature as shown in Figure 4.56 below.

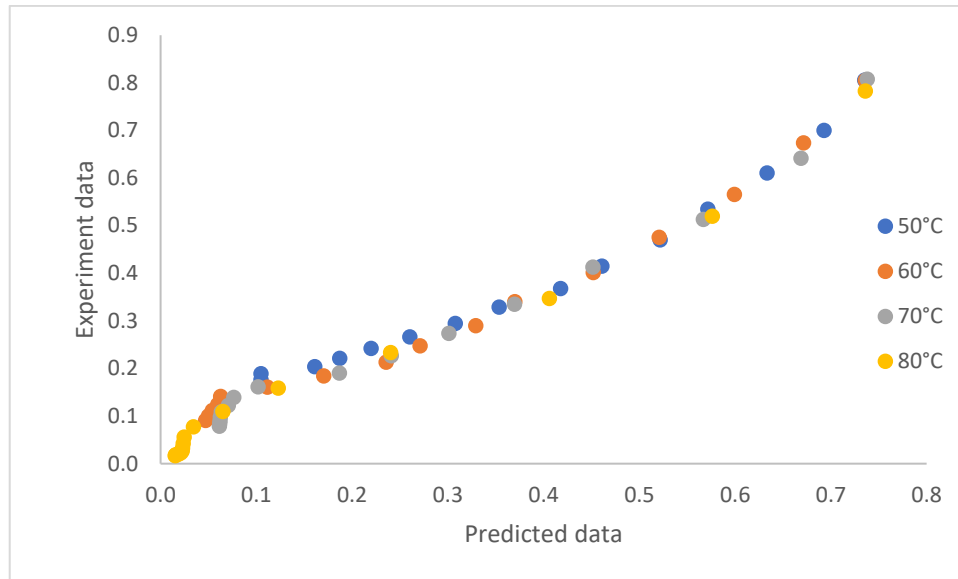


Figure 4.56: Comparison of experimental and predicted data (moisture content) by Simplified Fickian model

Figure 4.56 above shows that the generated trendline was linear from origin. This behavior indicates that the predicted model is a good fit for the actual drying data. Therefore, it is suggested that Simplified Fickian model can be used for the drying process prediction of sago starch. According to Inyang et al. (2018), simplified Fickian model will only show good data fitting with drying experiment that have same condition with the model drying condition during its development.

4.7.3 Assessment of Characteristic Drying Curve for Sago Starch Drying Kinetic

In this study, a normalized drying rate was developed for each drying method by using the functions suggested by Jannot et al. (2004) which was also used by Baini and Langrish, (2007). The fitted parameters were given as $a=0.079$, $b=2.541$, $c=0.478$, $d=-0.004$ and $N_v = 1.716 \text{ kg kg}^{-1}\text{s}^{-1}$. The results were then compared with the experimental data, as shown in Figure 4.57 to Figure 4.61 below.

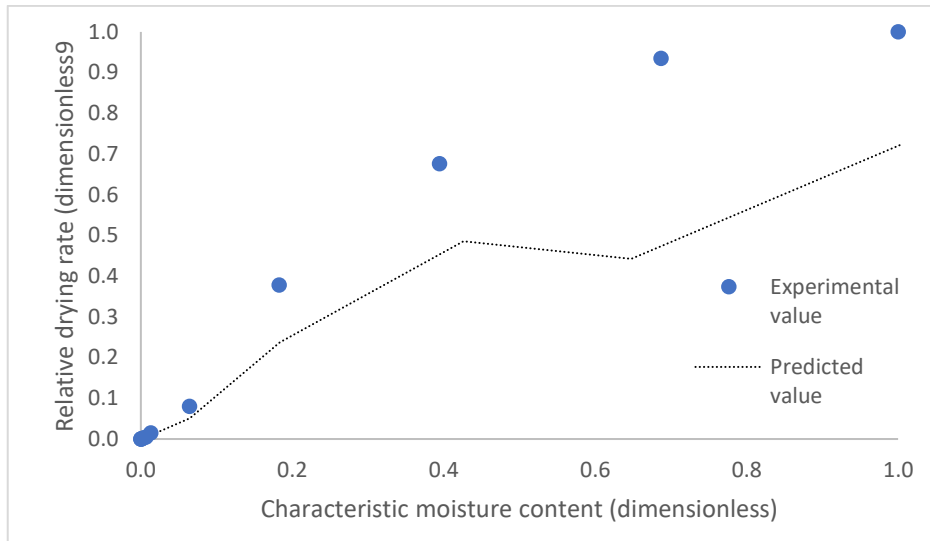


Figure 4.57: The relative drying rate as a function of the characteristic moisture content for continuous drying of sago starch

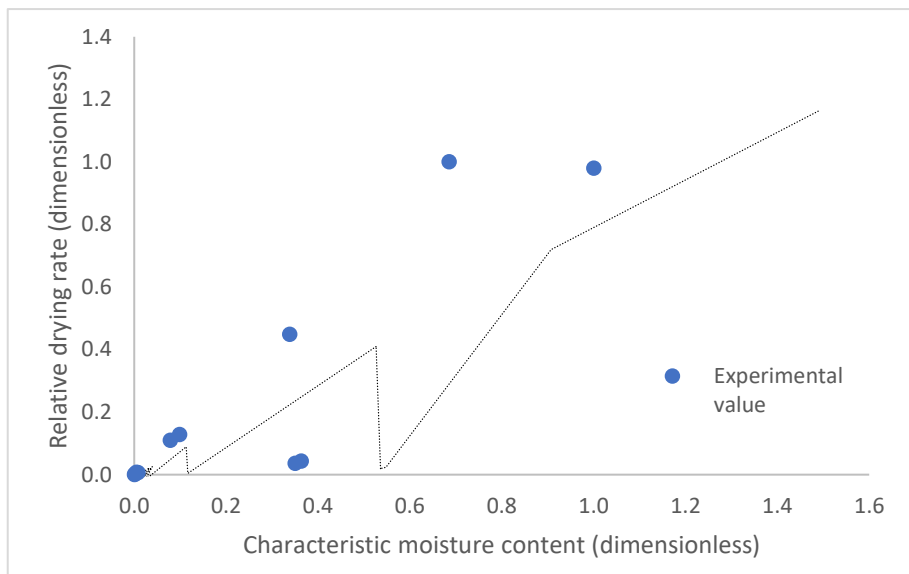


Figure 4.58: The relative drying rate as a function of the characteristic moisture content for intermittent drying of sago starch

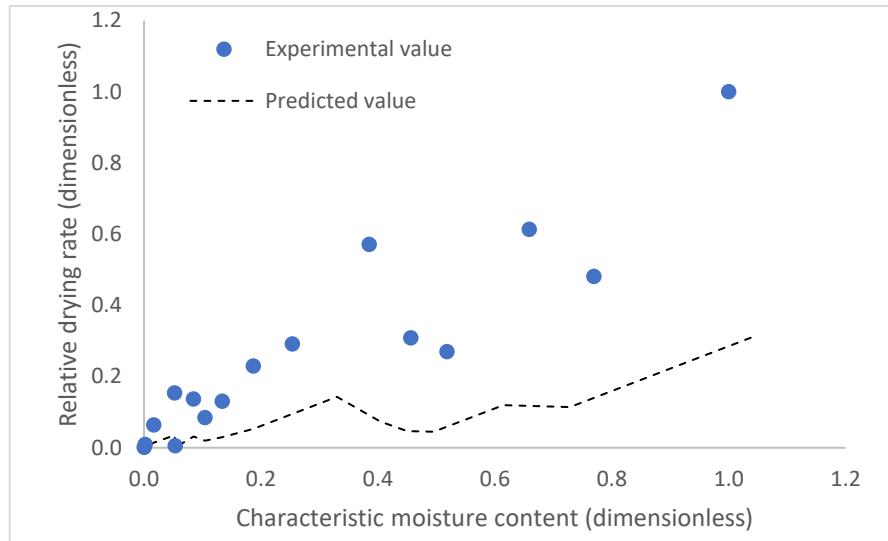


Figure 4.59: The relative drying rate as a function of the characteristic moisture content for sun drying of sago starch

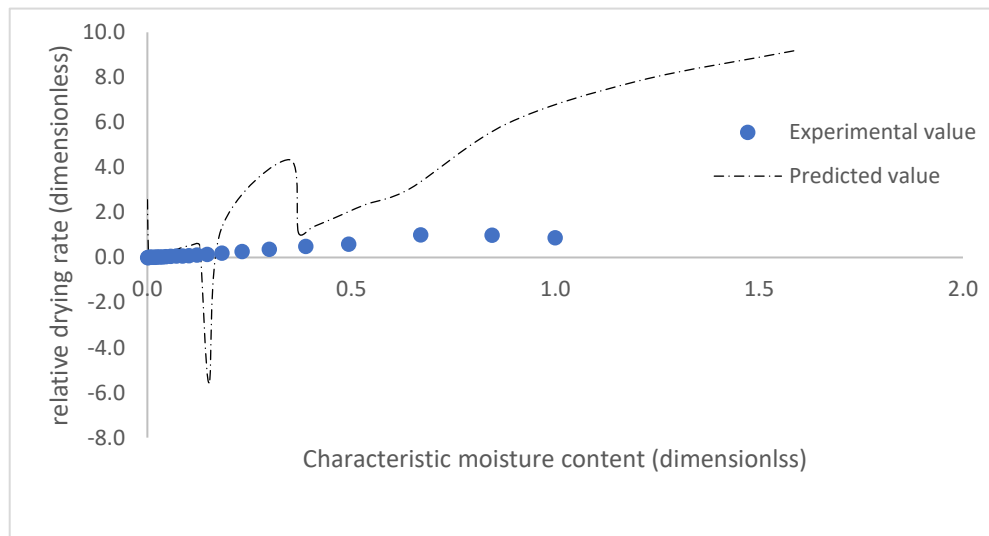


Figure 4.60: The relative drying rate as a function of the characteristic moisture content for microwave drying of sago starch

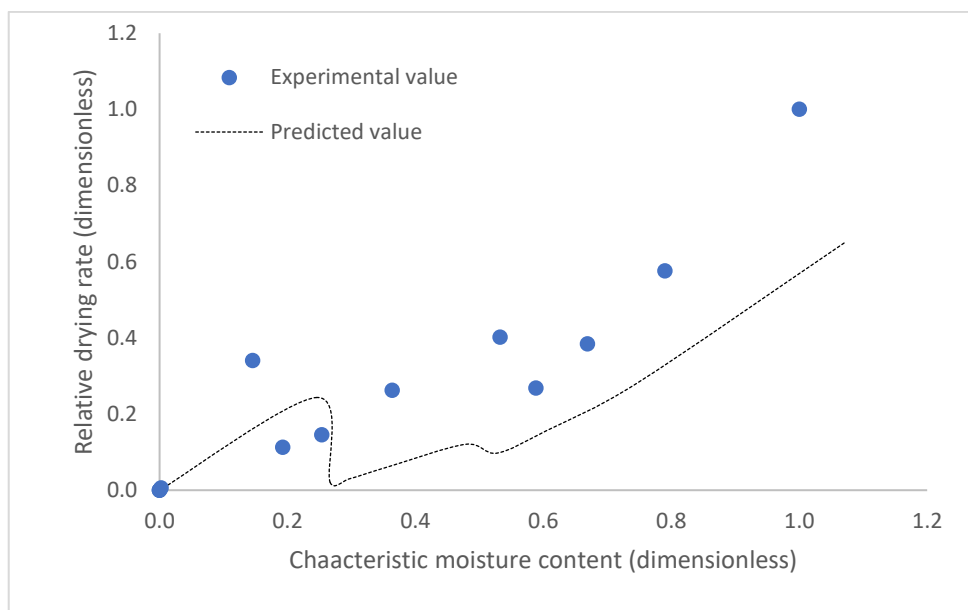


Figure 4.61: The relative drying rate as a function of the characteristic moisture content for freeze drying of sago starch

Figure 4.57 to Figure 4.61 show the relative drying rate as a function of the characteristic moisture content for different drying methods of sago starch. All figures show the different shape of normalized drying curve between experimental data and predicted value. In addition to that, the drying rates were scattered in all results. Therefore, it can be deducted that the characteristic drying curve concept may not be an appropriate approach to modeling any of the drying method of sago starch. Characteristic drying curve has been used for several foods and materials such as banana (Baini & Langrish, 2007; Jannot et al., 2004), oil palm fibers (Puspasari et al., 2014), spearmint (Ayadi et al., 2013), and citrus fruits fibers (Langrish, 2008). However, the model has not been used for starch yet, thus, it was shown as not suitable for sago starch drying, in this study.

4.7.4 Concluding Remarks

The results from this section show empirical model and Fickian model fitted into sago starch drying kinetic, while characteristic drying curve is not suitable for the purpose. Furthermore, the fitted drying parameter has been obtained from each drying temperature and method.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Based on the results it can be concluded that all the objectives set as explained in Chapter 1 is achieved and summarised as follows.

First objective, which relate to the characterization of sago starch, has been achieved. Properties that include the content of moisture, amylose, protein, lipid, ash, and analysis on resistant starch, granules morphology, particle size, color, viscosity, FTIR spectrum and thermal properties were successfully analyzed. Sarawak sago starch (lemantak) has slightly higher moisture content of 20% and lower whiteness index; however, it has higher starch, resistant starch content, and viscosity value, bigger granule size with high crystalline characteristic indicating its better quality compared to sago flour produced commercially.

Second objective was to identify the differences in properties of sago starch samples that were dried to different final moisture content, has been achieved. Drying of fresh sago starch showed that it can be dried up to 10% m.c. (d.b) without degrading the starch and resistant starch content, no changes in morphology, granule size and thermal stability to the extent of 300°C. Interestingly, sago starch with lower moisture content has higher whiteness index and lower viscosity value that is good for industrial acceptance. The results confirmed that a combining method of manual extraction and drying process is reliable to produce better quality of sago starch, but further study is required to identify a suitable drying method and condition.

The third objective was to study the Equilibrium Moisture Content (EMC), moisture diffusivities and drying kinetics of sago starch by using different drying methods, variables, and parameter, has been achieved. Equilibrium moisture content is highly affected by drying temperature, followed by air velocity, sample thickness, position, and drying method. Drying temperature has a great influence on the drying rate of sago starch, while air velocity and sample thickness have an effect to a certain extent. The highest whiteness index of dried sago starch is obtained at drying temperature of 60°C and air velocity of 0.965m/s. Microwave drying produce the highest moisture diffusivities that shows radiation caused faster moisture movement. It was found that the diffusivity of sago starch significantly influenced by drying temperature. However, other variable such as air velocity, sample thickness and position increase the moisture diffusivity to a certain extent.

The fourth objective was to assess the drying models for sago starch through data fitting several empirical models, a simplified Fickian model and the Characteristic Drying Curve model, has been achieved. Analysis of models showed that different types if empirical models fit in different drying condition. Continuous and sun drying can be predicted by using same models that are Lewis Newton, Page, Henderson & Pabis, Modified Henderson & Pabis, Wang & Singh, Approximation of diffusion, and Verma. All models except Wang & Singh model can predict the drying kinetic for intermittent drying during its tempering period. However, all models show poor fitting for freeze drying. In contrast, all models can be used to predict drying kinetic of sago starch from microwave drying. Meanwhile, simplified Fickian model were able to predict sago starch dried from 50 to 80°C. However, the application of characteristic drying curve concept was found to be not appropriate to be used for sago starch drying.

5.2 Recommendations

Recommendation of further study are on the following areas;

5.2.1 Characterization

As for properties, study on gel strength shall be done since it will give insight on textural properties of sago starch end-product. As for experimental analysis, it is suggested to run the X-ray diffraction (XRD) analysis to get a brief idea on sago starch integrity changes upon drying.

5.2.2 Kinetics

As for the drying kinetic study, since the humidity control cannot be applied in this study, it is suggested that this variable to be included in the future. It is also recommended to conduct a finite element analysis so that the moisture diffusion inside the sago starch granule can be simulated. Finally, this study hopes that the results will be useful to be applied in the drying machine of sago starch.

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APPENDICES

LIST OF APPENDICES

No.	Title
A	Journal Publications
B	Conference Proceedings
C	Photos
D	Calculation of Air Velocity
E	ANOVA and Post-hoc t-test results
F	Arrhenius plot
G	Model fitting confirmation graph

Appendix A: Journal Publications

1. “Effect of temperature to the properties of Lemantak”, **M. Mustafa Kamal**, R. Baini, S.Mohamaddan, O. S. Selaman, N. Ahmad Zauzi, M. R. Rahman, N. Abdul Rahman, Chong, K.H., M.F. Atan, N.A.S. Abdul Samat, S.N.L.Taib, A.K.Othman (2017), 28th Symposium of Malaysian Chemical Engineers (SOMChE) 2016, IOP Conf. Series: Materials Science and Engineering **206** (2017) 012-039.
2. “Comparisons of the physicochemical and functional properties of commercially and traditionally processed sago starch”, **Kamal, M. M.**, Baini, R., Mohamaddan, S., Ragai, A. R. H., Fong, L. S., Rahman, N. A., Taib, S. N. L. (2017). , MATEC Web of Conference, 87, 03007, 0–5.
3. **Kamal, M. M.**, Baini, R., Lim, S. F., Rahman, M. R., Mohamaddan, S., & Hussain, H. (2019). Drying effect on the properties of traditionally processed sago starch. International Food Research Journal, 26(2), 1861–1869.
4. **Kamal M.M.**, Baini R., Fong L.S., Hussain M.H.M., Mohamaddan S. (2020) Effect of Drying Temperature to the Thin Layer Drying Model of Sago Starch. In: Reddy A., Marla D., Simic M., Favorskaya M., Satapathy S. (eds) Intelligent Manufacturing and Energy Sustainability. Smart Innovation, Systems and Technologies, vol 169. Springer, Singapore.

Appendix B: Conference Proceedings

1. “Effect of temperature to the properties of sago starch”, **M. Mustafa Kamal**, R. Baini, S.Mohamaddan, O. S. Selaman, N. Ahmad Zauzi, M. R. Rahman, N. Abdul Rahman, Chong, K.H., M.F. Atan, N.A.S. Abdul Samat, S.N.L.Taib, A.K.Othman, Proceedings of the 29th Symposium of Malaysian Chemical Engineers (SOMChE) 2016, Miri, Sarawak, Malaysia, December 1 – 3rd, 2016.
2. “Comparisons of the physicochemical and functional properties of commercially and traditionally processed sago starch”, **M.Mustafa Kamal**, R.Baini, S.Mohamaddan, A.R.Henry Ragai, L. Soh Fong, N. Abdul Rahman, N. Mili, S.N.L.Taib, A.K.Othman, M.O. Abdullah2, International UNIMAS STEM 9th Engineering Conference 2016 (EnCon2016), Riverside Majestic, Kuching, Sarawak, August 26-28th, 2016.
3. “Roles of Moisture Content in Lemantak (traditional wet sago starch)”, **M. Mustafa Kamal** & R. Baini,, 5th Postgraduate Borneo Research Colloquium 2017, Center for Training Facilities, Universiti Malaysia Sarawak, 4 - 6th July 2017.

Appendix C: Photos



Figure 1 Shimadzu Fourier Transform Infrared (FTIR) IRAFFINITY-1

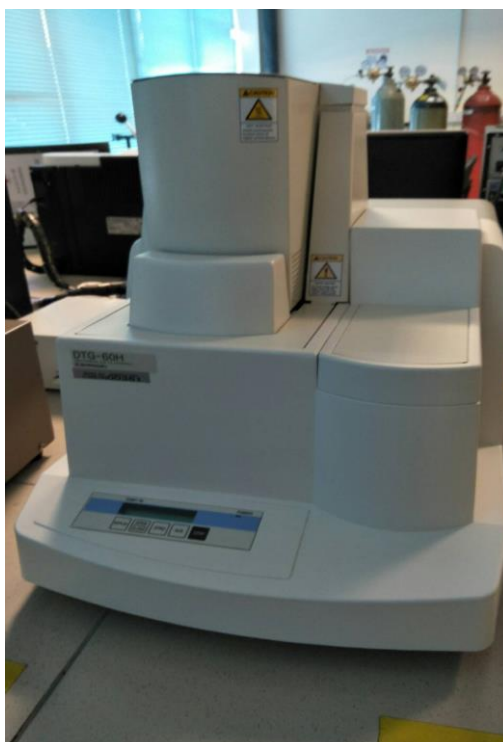


Figure 2: SHIMADZU DTG-60 H



Figure 3: Freeze dryer

Appendix D: Calculation of Air Velocity

The air velocity for drying experiment was calculated based on the ACH formula below.

$$ACH = Q / Vol \quad \text{Equation 7.1}$$

ACH is the number of air changes per hour (x/h), and the given ACH for the oven model was 9 x/h or 0.15 x/min in 100% of fan speed. In which, Q is the volumetric flow rate of air in cubic feet per minute (cfm), while Vol is the space volume, L * W * H in cubic feet (L = Length, W = Width and H = Height). According to the oven internal dimension, the space volume was calculated as, Vol = 1.6893(L) * 2.5591(W) * 2.1326(H), that made up to 9.219 cubic feet. The Q was then determined as, Q = 0.15 (air changes per hour) x 9.219 (cubic feet) that is equal to 1.383 cfm.

From the volume flow (cfm), the air velocity in feet per minute (fpm) can be calculated by divided the cfm with the cross-section area of duct (πr^2). Therefore,

$$\text{Air velocity (fpm)} = \text{Air flow (cfm)} / \text{Area } (\pi r^2) \quad \text{Equation 7.2}$$

Given that $V = 1.383 / (\pi \times 26^2)$. Therefore, $V = 189.97$ fpm. The air velocity was then converted to SI unit of meter per second as 0.97 m/s which is refer to 100% of fan speed. The fan speed was set to 0, 40, 70 and 100%. Therefore, the air velocity for each of fan speed of 0, 40, 70 and 100% was calculated as 0, 0.386, 0.676 and, 0.965 m/s respectively.

Appendix E: ANOVA and Post-hoc t-test results

Table 1: ANOVA and Post-hoc t-test results of L value from different sago starch moisture content. Significance value was $p < 0.05$

Sample	MC40	MC30	MC20	MC10		
Data 1	61.77	79.4	82.74	85.73		
Data 2	61.05	79.22	82.33	85.31		
Data 3	61.22	79.31	82.41	85.44		
Mean	61.35	79.31	82.49	85.49		
Standard Deviation	0.38	0.09	0.22	0.22		
P value (ANOVA)	1.10E-13					
t-test Pair	MC40 vs MC30	MC40 vs MC20	MC40 vs MC10	MC30 vs MC20	MC30 vs MC10	MC20 vs MC10
P value (t-test)	1.43E-07	1.19E-07	6.92E-08	1.96E-05	1.34E-06	7.03E-05

Table 2: ANOVA and Post-hoc t-test results of a value from different sago starch moisture content. Significance value was $p < 0.05$

Sample	MC40	MC30	MC20	MC10		
Data 1	15.33	3.4	1.23	0.04		
Data 2	15.32	3.15	1.24	0.05		
Data 3	15.41	3.16	1.25	0.03		
Mean	15.35	3.24	1.24	85.49		
Standard Deviation	0.05	0.14	0.01	0.01		
P value (ANOVA)	2.60E-16					
t-test Pair	MC40 vs MC30	MC40 vs MC20	MC40 vs MC10	MC30 vs MC20	MC30 vs MC10	MC20 vs MC10
P value (t-test)	1.56E-08	1.08E-10	7.78E-11	1.68E-05	2.58E-06	1.29E-08

Table 3: Post-hoc t-test results of b value from different sago starch moisture content. Significance value was $p < 0.0125$

Sample	MC40	MC30	MC20	MC10		
Data 1	9.65	8.71	6.79	5.12		
Data 2	9.67	8.73	6.78	5.13		
Data 3	9.64	8.72	6.77	5.11		
Mean	9.65	8.72	6.78	5.12		
Standard Deviation	0.02	0.01	0.01	0.01		
P value (ANOVA)	1.72E-18					
t-test Pair	MC40 vs MC30	MC40 vs MC20	MC40 vs MC10	MC30 vs MC20	MC30 vs MC10	MC20 vs MC10
P value (t-test)	9.75E-08	1.09E-09	1.75E-10	1.88E-09	1.59E-10	3.51E-09

Table 4: ANOVA and Post-hoc t-test results of WI value from different sago starch moisture content. Significance value was $p < 0.05$

Sample	MC40	MC30	MC20	MC10		
Data 1	57.69	77.41	81.41	84.84		
Data 2	57.65	77.4	81.42	84.96		
Data 3	57.66	77.43	81.4	84.32		
Mean	57.67	77.41	81.41	84.71		
Standard Deviation	0.02	0.02	0.01	0.34		
P value (ANOVA)	2.36E-15					
t-test Pair	MC40 vs MC30	MC40 vs MC20	MC40 vs MC10	MC30 vs MC20	MC30 vs MC10	MC20 vs MC10
P value (t-test)	1.95E-12	5.97E-13	1.68E-08	2.90E-10	3.15E-06	7.40E-05

Table 5: ANOVA and Post-hoc t-test results of Peak Viscosity (AU) value from different sago starch moisture content. Significance value was $p < 0.05$

Sample	MC40	MC30	MC20	MC10		
Data 1	659	621	607	575		
Data 2	658	622	613	569		
Data 3	660	625	601	577		
Mean	659.00	623	607	574.00		
Standard Deviation	1.00	2.08	6.00	4.16		
P value (ANOVA)	2.74E-08					
t-test Pair	MC40 vs MC30	MC40 vs MC20	MC40 vs MC10	MC30 vs MC20	MC30 vs MC10	MC20 vs MC10
P value (t-test)	1.08E-05	1.21E-04	4.20E-06	1.29E-02	5.32E-05	1.38E-03

Appendix F: Arrhenius plot

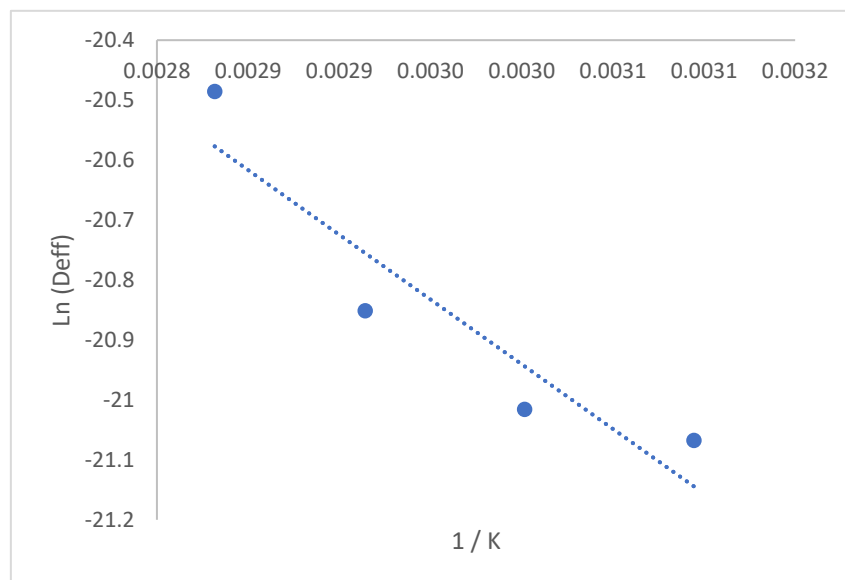


Figure 1: Arrhenius plot for $\ln(D_{eff})$ vs inverse of temperature for drying sago starch in different temperature

Appendix F: Model fitting confirmation graph

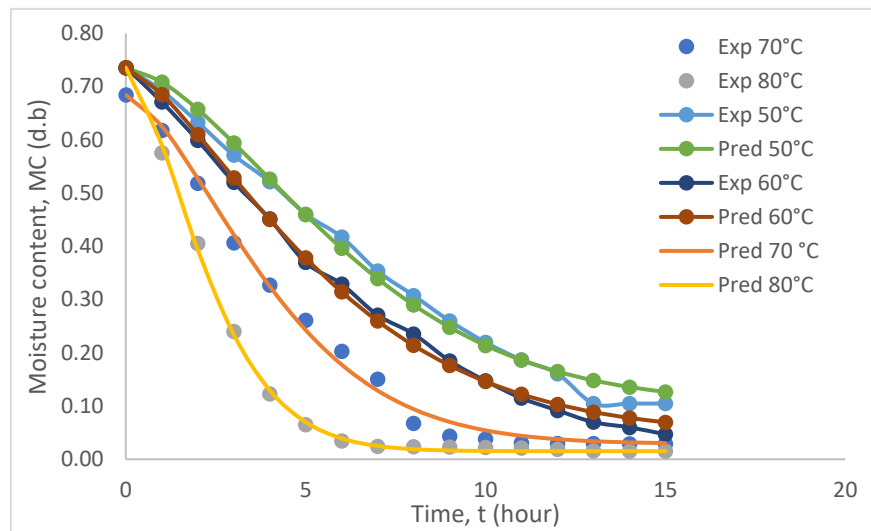


Figure 1: Predicted and experimental value of sago starch moisture kinetic through Page model for different drying temperature of 50, 60, 70 and 80 °C

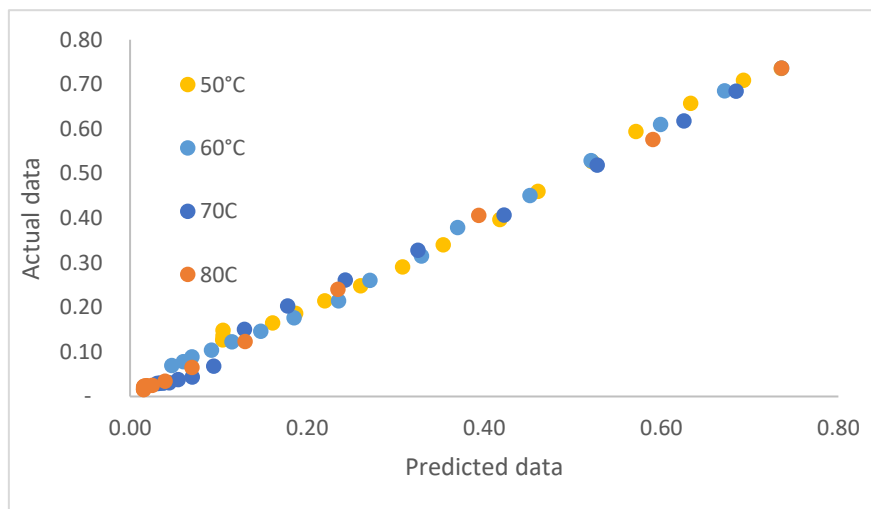


Figure 2: Linear plot of sago starch experimental and predicted value through Page model for different drying temperature of 50, 60, 70 and 80 °C

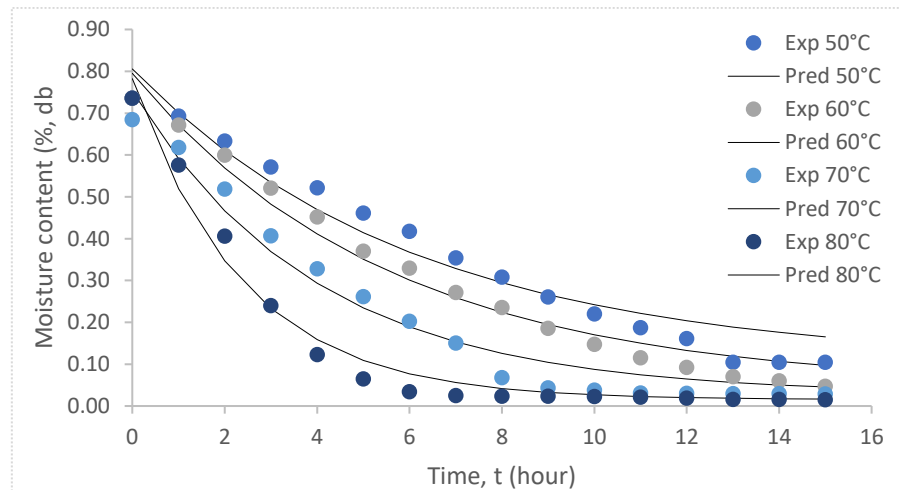


Figure 3: Predicted and experimental value of sago starch moisture kinetic through Henderson & Pabis model for different drying temperature of 50, 60, 70 and 80 °C

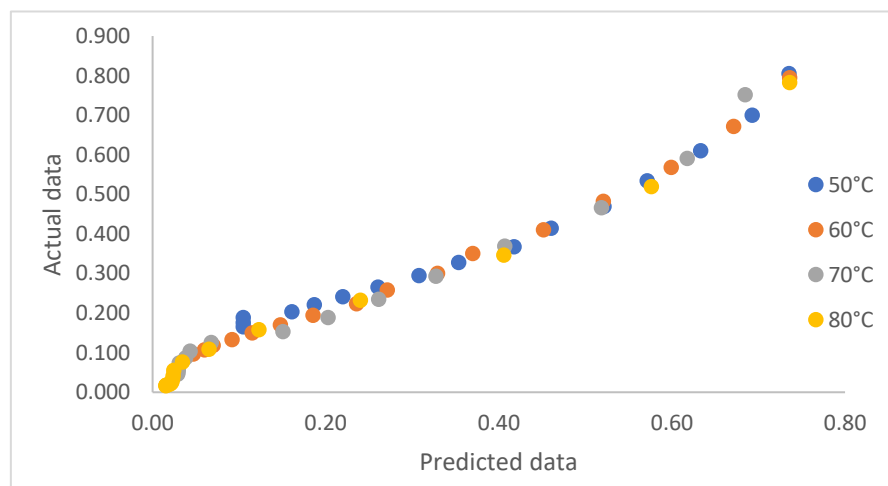


Figure 4: Linear plot of sago starch experimental and predicted value through Henderson & Pabis model for different drying temperature of 50, 60, 70 and 80 °C

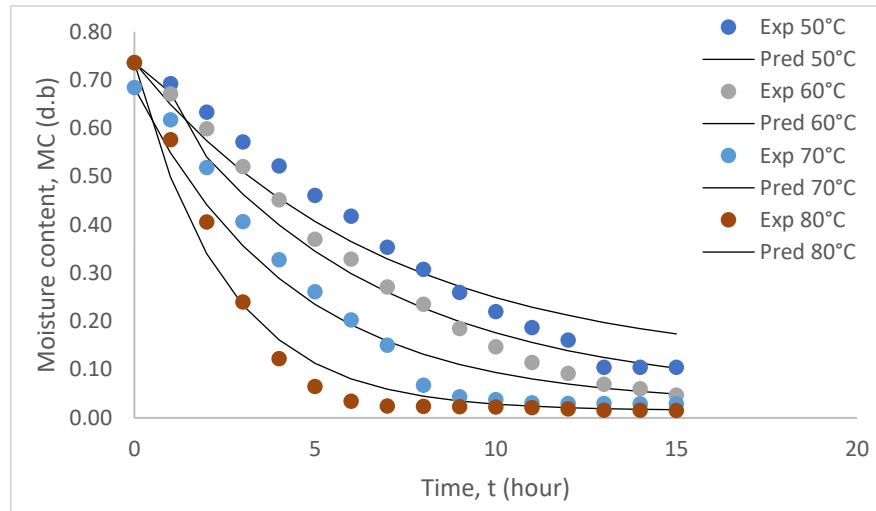


Figure 5: Predicted and experimental value of sago starch moisture kinetic through Modified Henderson and Pabis model for different drying temperature for 50, 60, 70, and 80 °C

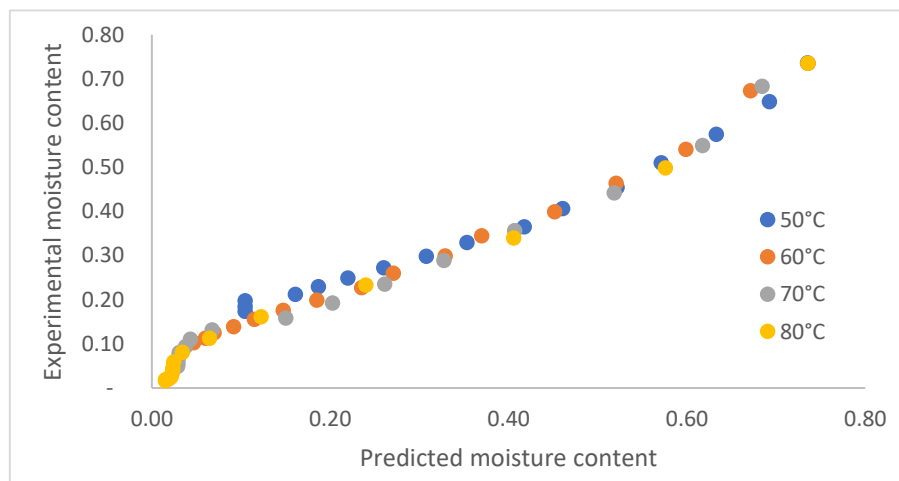


Figure 6: Linear plot of sago starch experimental and predicted value through Modified Henderson & Pabis model for different drying temperature of 50, 60, 70 and 80 °C

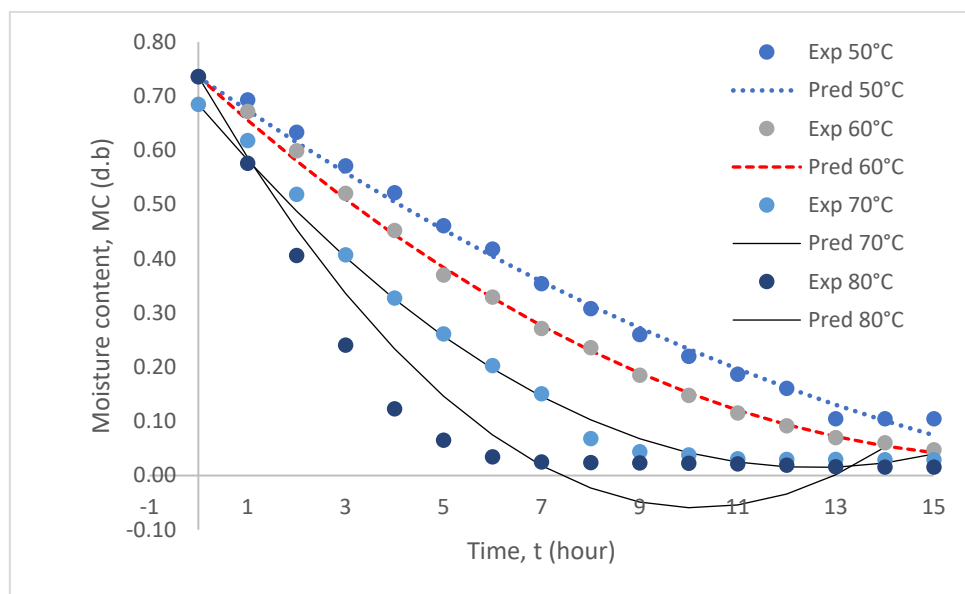


Figure 7: Predicted and experimental value of sago starch moisture kinetic through Wang & Singh model for different drying temperature for 50, 60, 70, and 80 °C

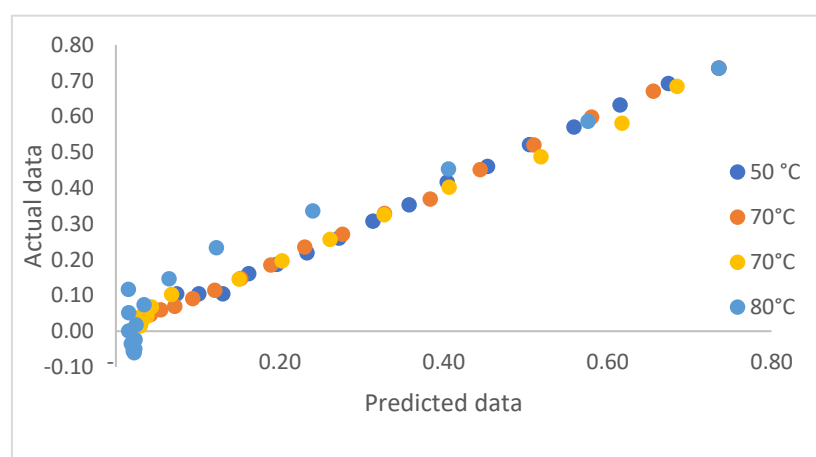


Figure 8: Linear plot of sago starch experimental and predicted value through Wang & Singh model for different drying temperature of 50, 60, 70 and 80 °C

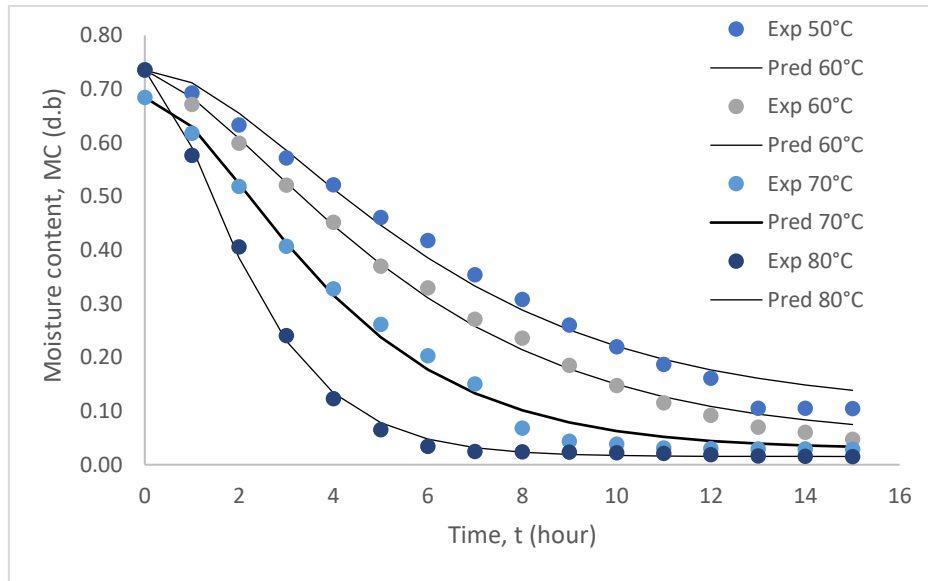


Figure 9: Predicted and experimental value of sago starch moisture kinetic through Approximation of Diffusion model for different drying temperature of 50, 60, 70 and 80 °C

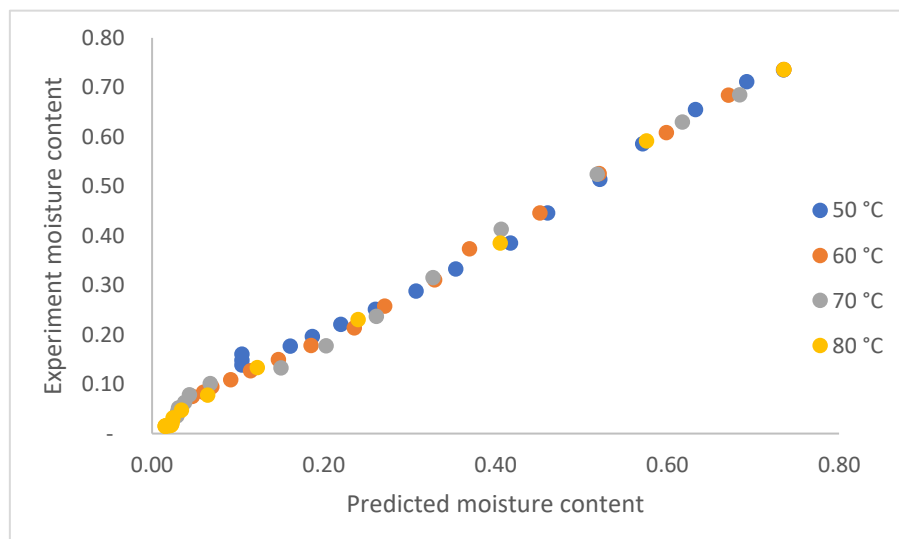


Figure 10: Linear plot of sago starch experimental and predicted value through Approximation of Diffusion model for different drying temperature of 50, 60, 70 and 80 °C

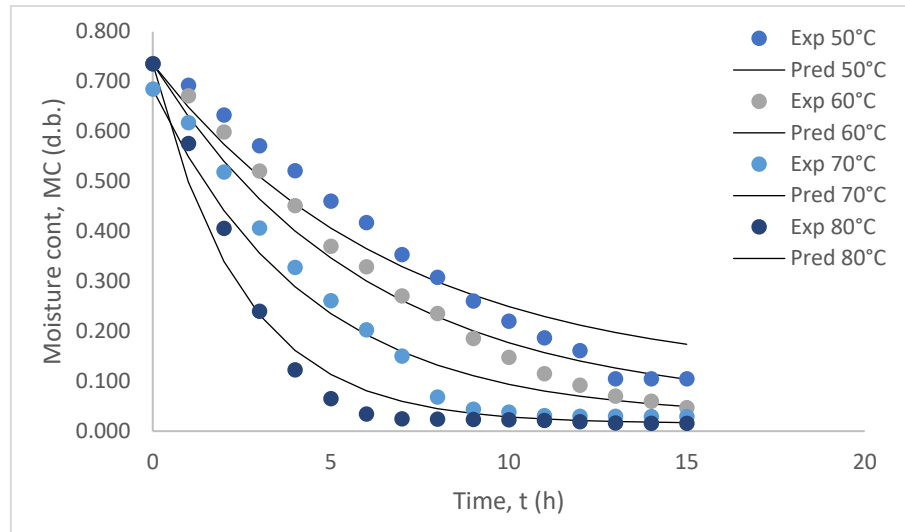


Figure 11: Predicted and experimental value of sago starch moisture kinetic through Lewis Newton model for different drying temperature for 50, 60, 70, and 80 °C

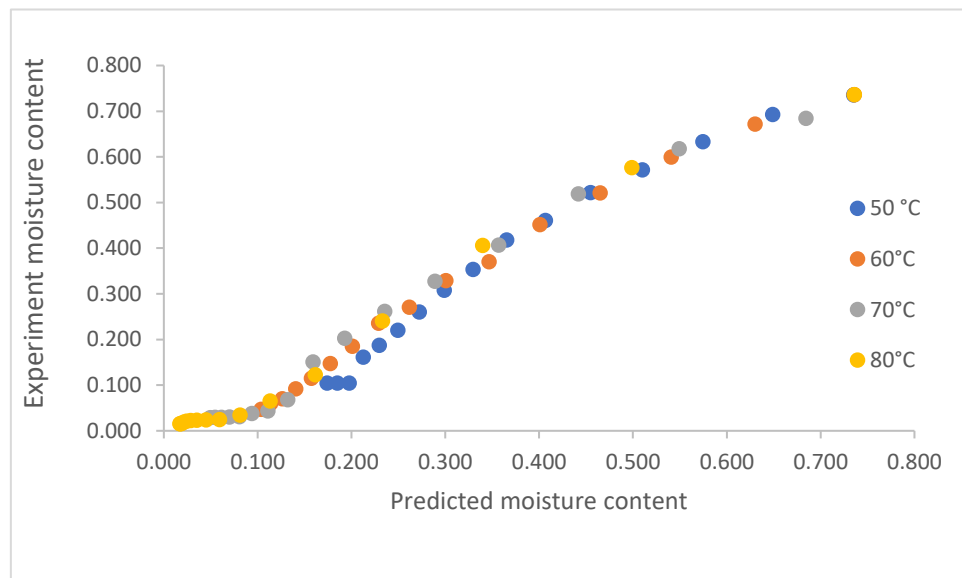


Figure 12: Linear plot of sago starch experimental and predicted value through Lewis Newton model for different drying temperature of 50, 60, 70 and 80 °C

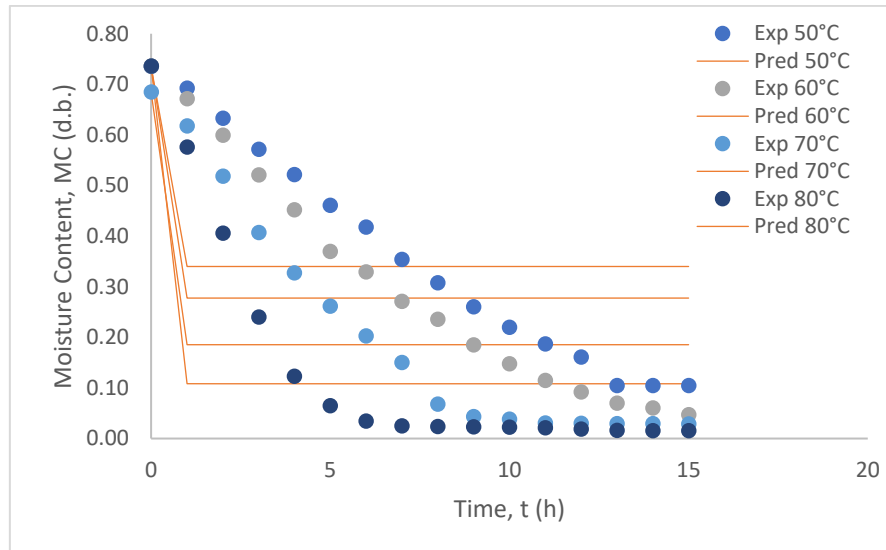


Figure 13: Predicted and experimental value of sago starch moisture kinetic through Logarithmic model for different drying temperature for 50, 60, 70, and 80 °C

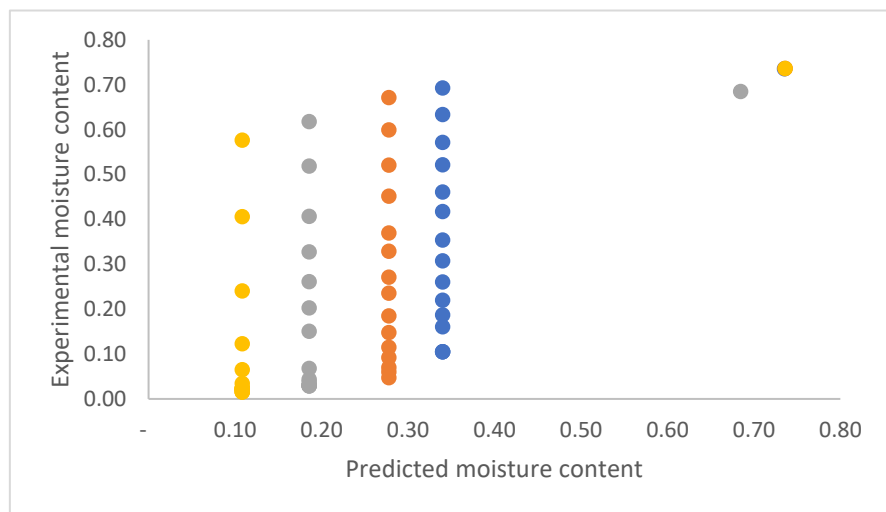


Figure 14: Linear plot of sago starch experimental and predicted value through Logarithmic model for different drying temperature of 50, 60, 70 and 80 °C

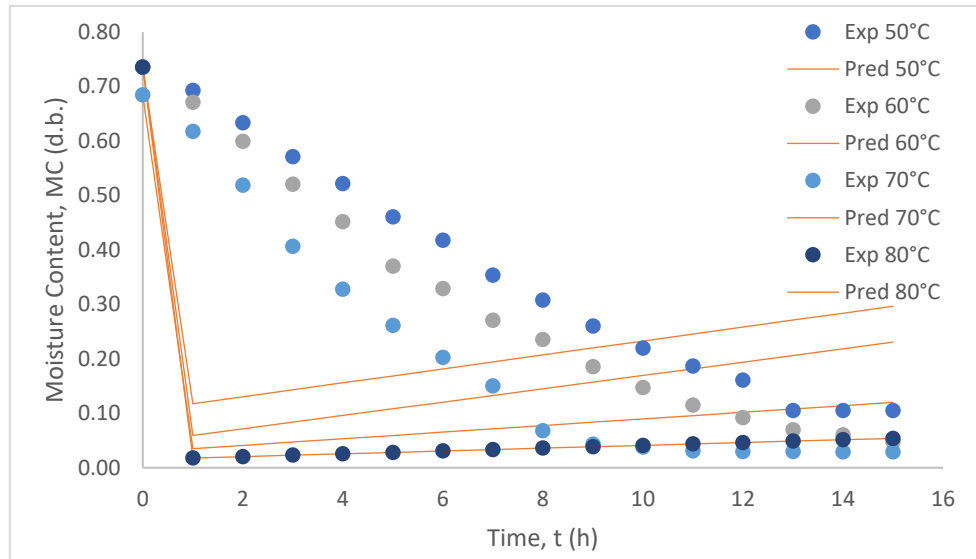


Figure 15: Predicted and experimental value of sago starch moisture kinetic through Midili Kucuk model for different drying temperature for 50, 60, 70, and 80 °C

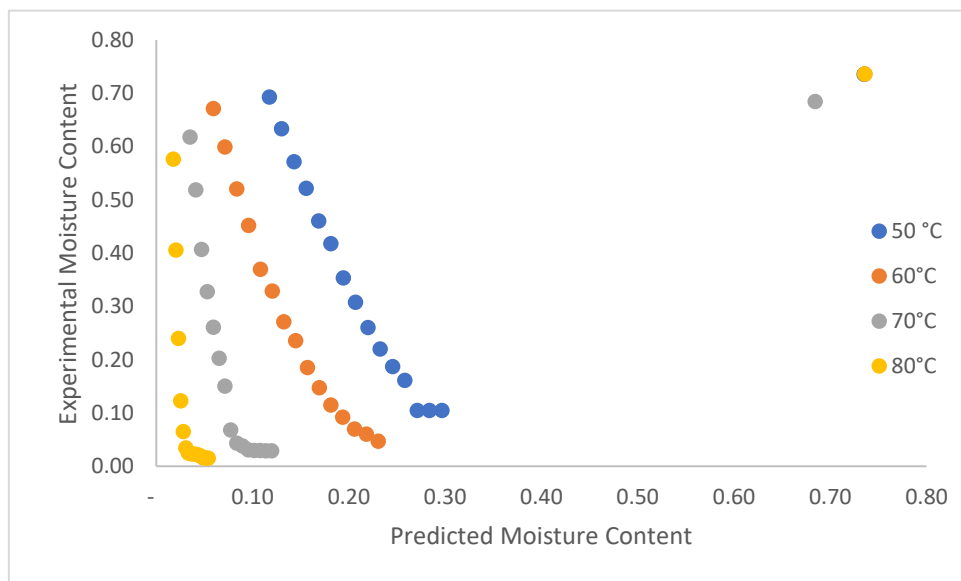


Figure 16: Linear plot of sago starch experimental and predicted value through Midili Kucuk model for different drying temperature of 50, 60, 70 and 80 °C

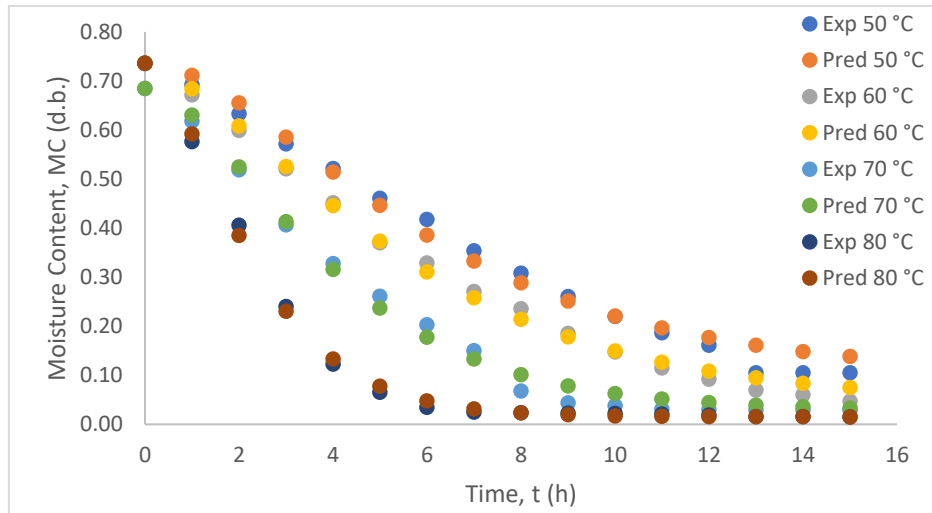


Figure 17: Predicted and experimental value of sago starch moisture kinetic through Verma model for different drying temperature for 50, 60, 70, and 80 °C

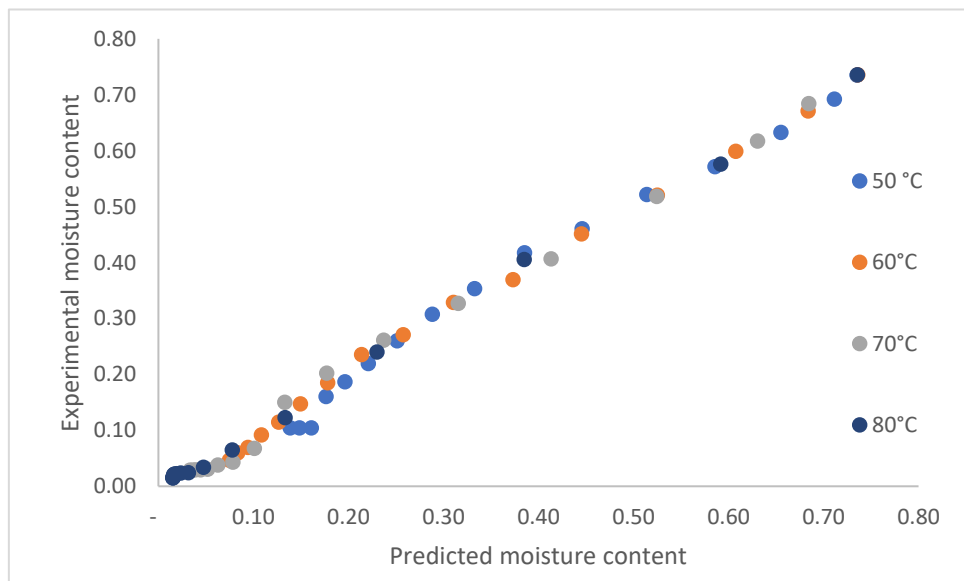


Figure 18: Linear plot of sago starch experimental and predicted value through Verma model for different drying temperature of 50, 60, 70 and 80 °C

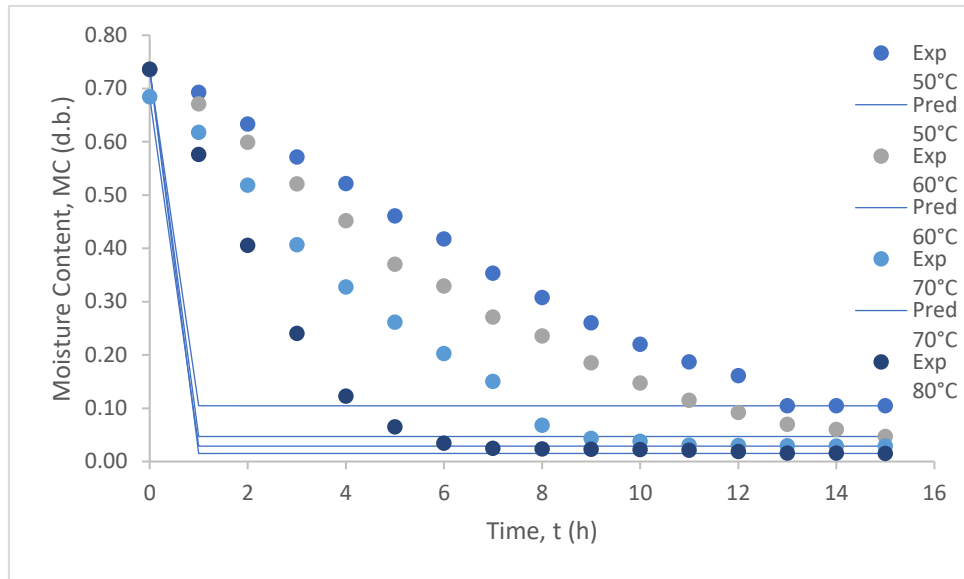


Figure 19: Predicted and experimental value of sago starch moisture kinetic through Two-Term model for different drying temperature for 50, 60, 70, and 80 °C

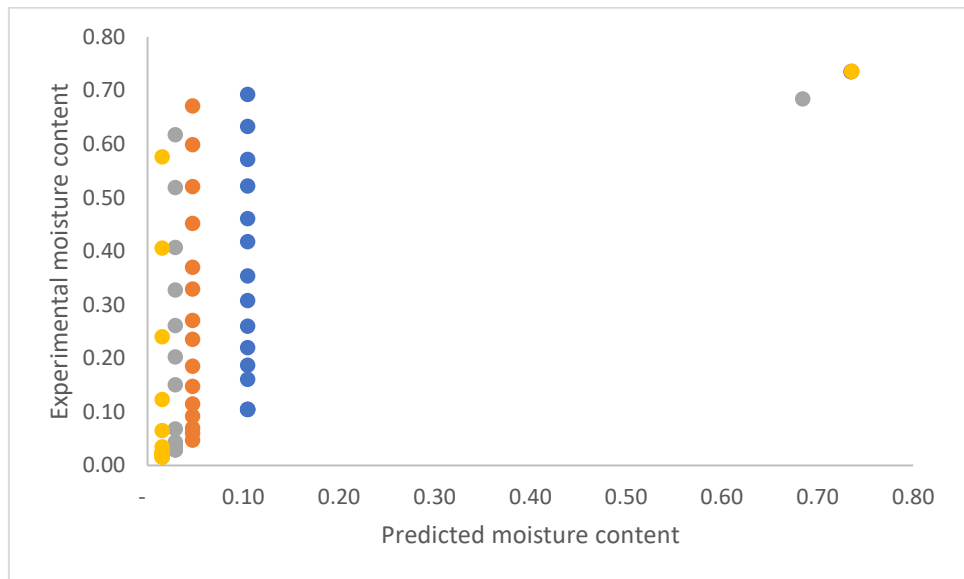


Figure 20: Linear plot of sago starch experimental and predicted value through Two-Term model for different drying temperature of 50, 60, 70 and 80 °C

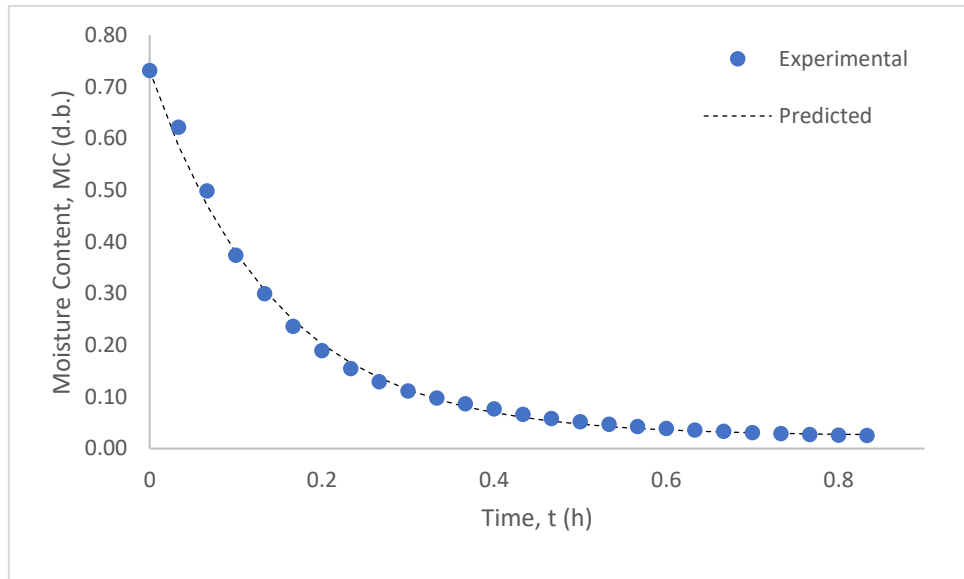


Figure 21: Predicted and experimental moisture content of sago starch using Lewis Newton model for microwave drying

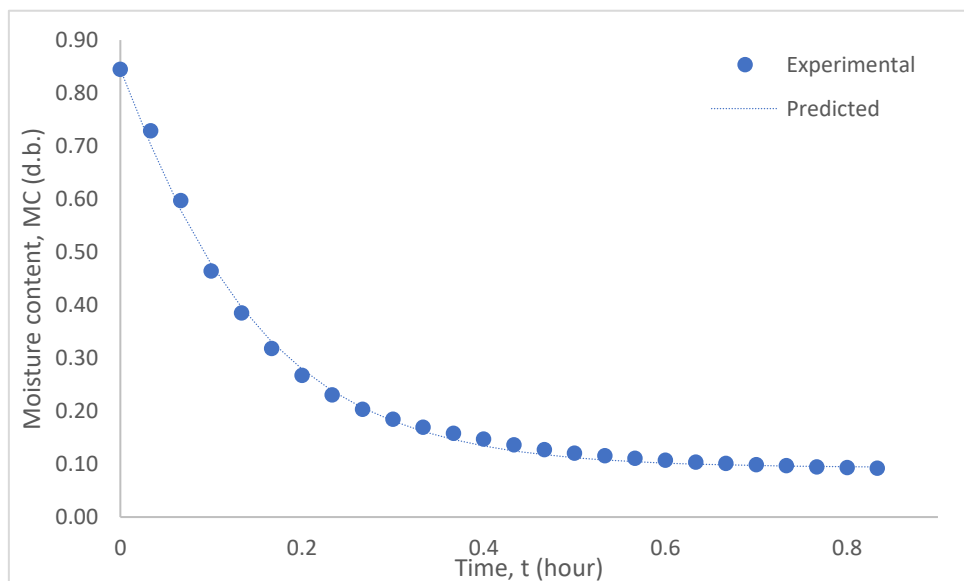


Figure 22: Predicted and experimental moisture content of sago starch using Page model for microwave drying

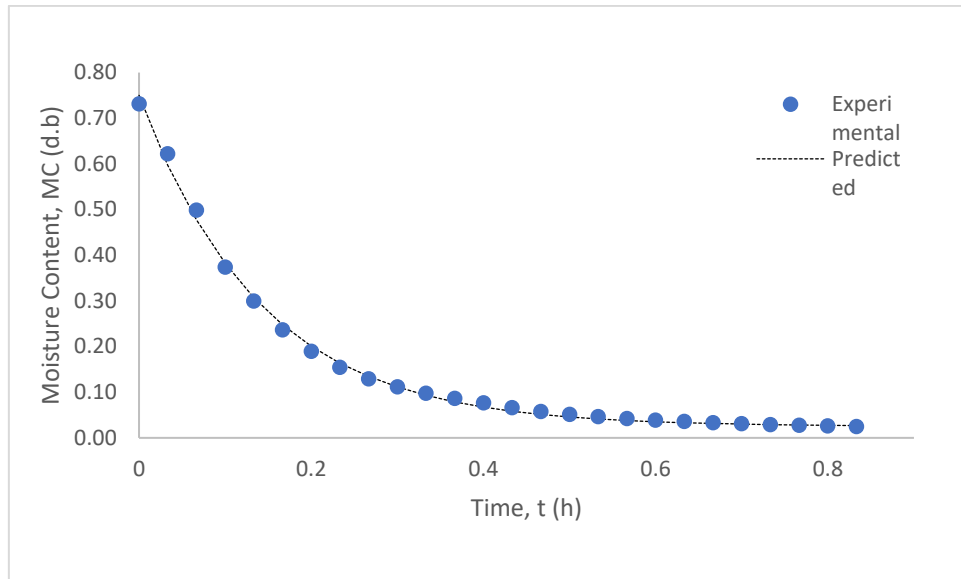


Figure 23: Predicted and experimental moisture content of sago starch using Henderson & Pabis model for microwave drying

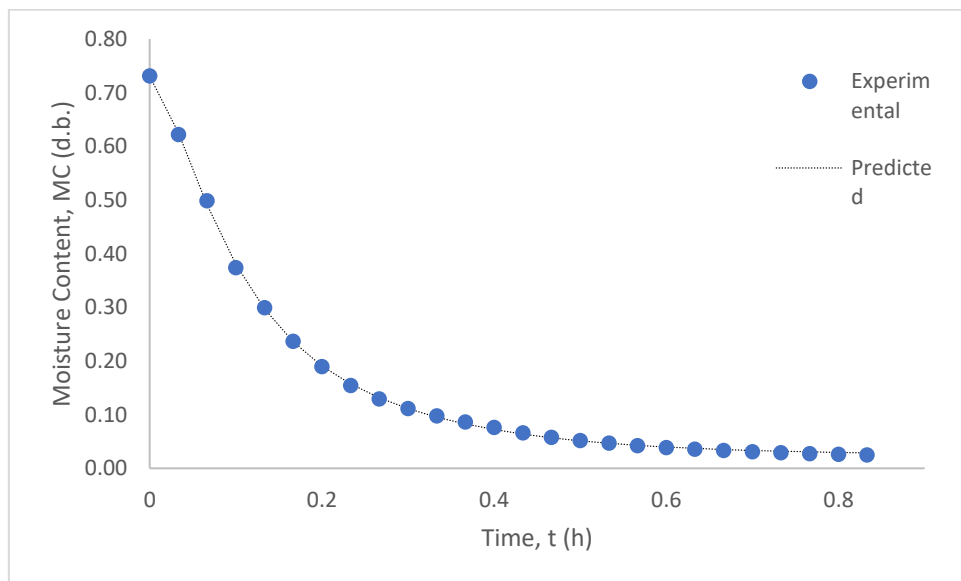


Figure 24: Predicted and experimental moisture content of sago starch using Modified Henderson & Pabis model for microwave drying

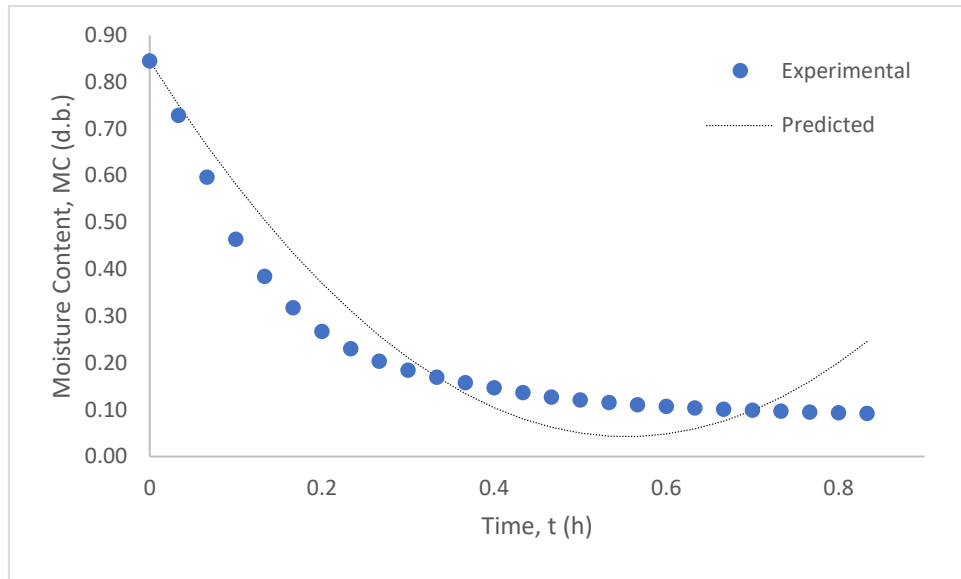


Figure 25: Predicted and experimental moisture content of sago starch using Wang & Singh model for microwave drying

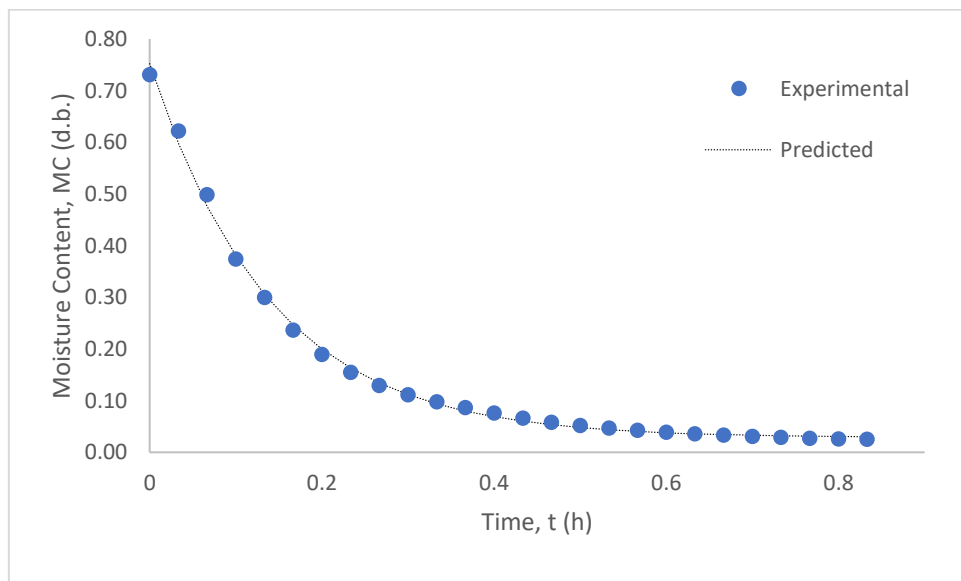


Figure 26: Predicted and experimental moisture content of sago starch using Logarithmic model for microwave drying

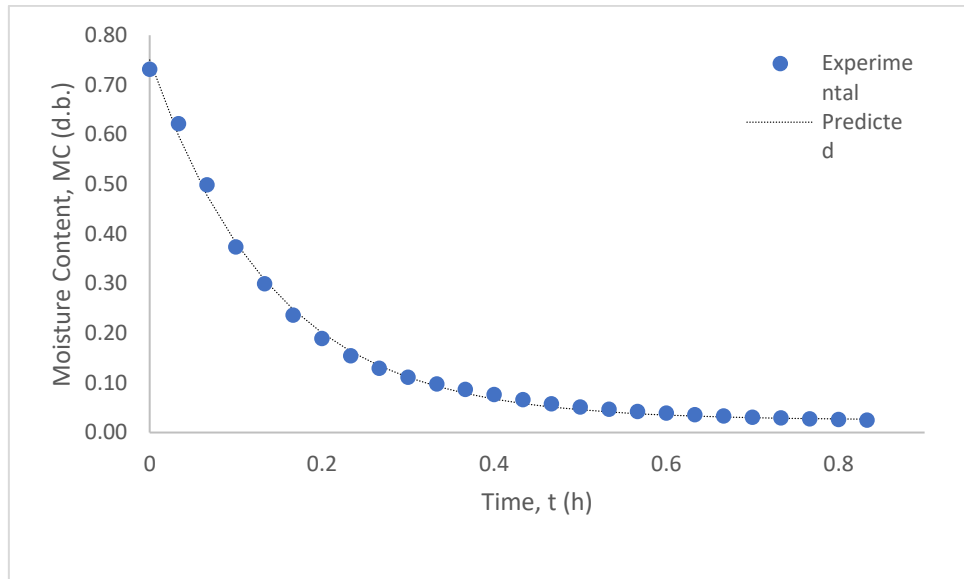


Figure 27: Predicted and experimental moisture content of sago starch using Two Term model for microwave drying

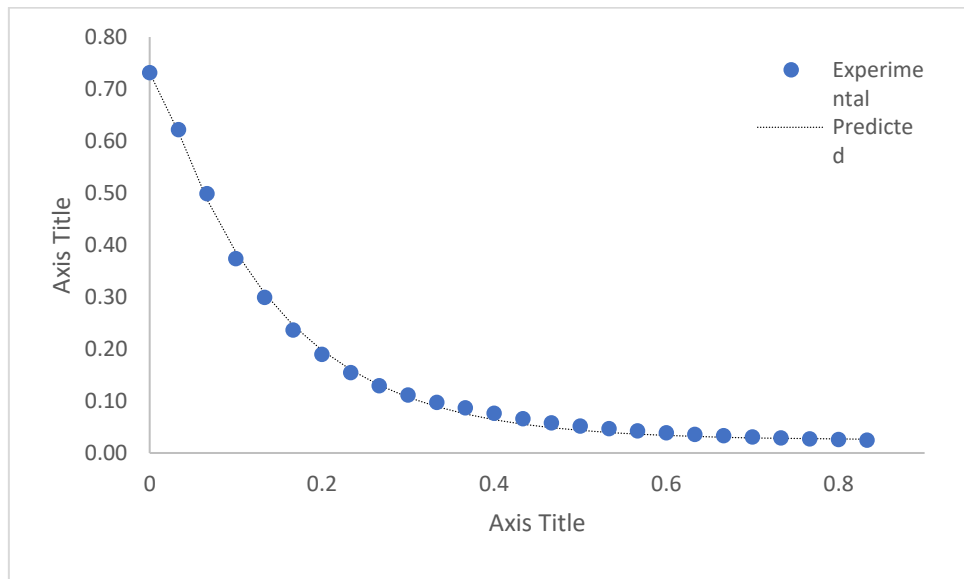


Figure 28: Predicted and experimental moisture content of sago starch using Approximation of Diffusion model for microwave drying

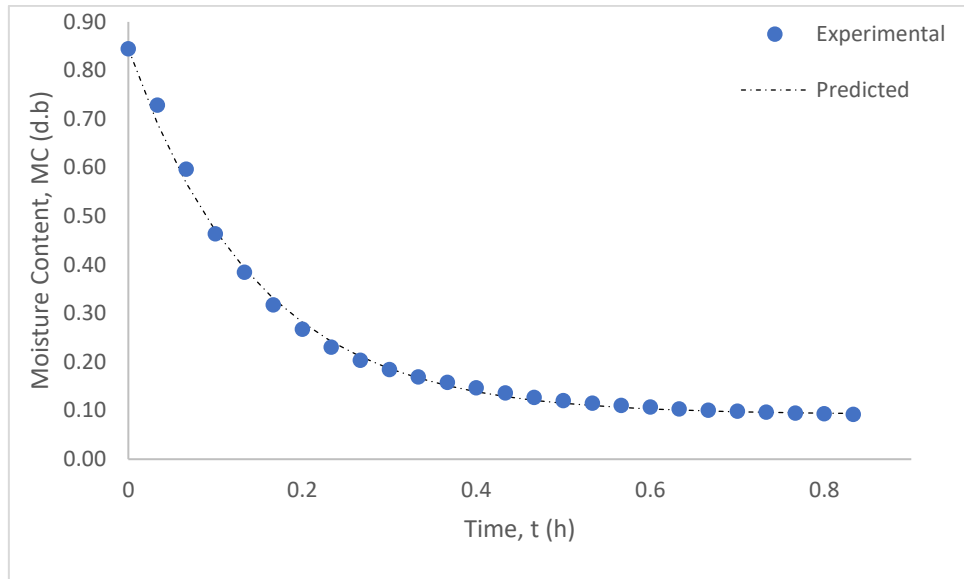


Figure 29: Predicted and experimental moisture content of sago starch using Verma model for microwave drying

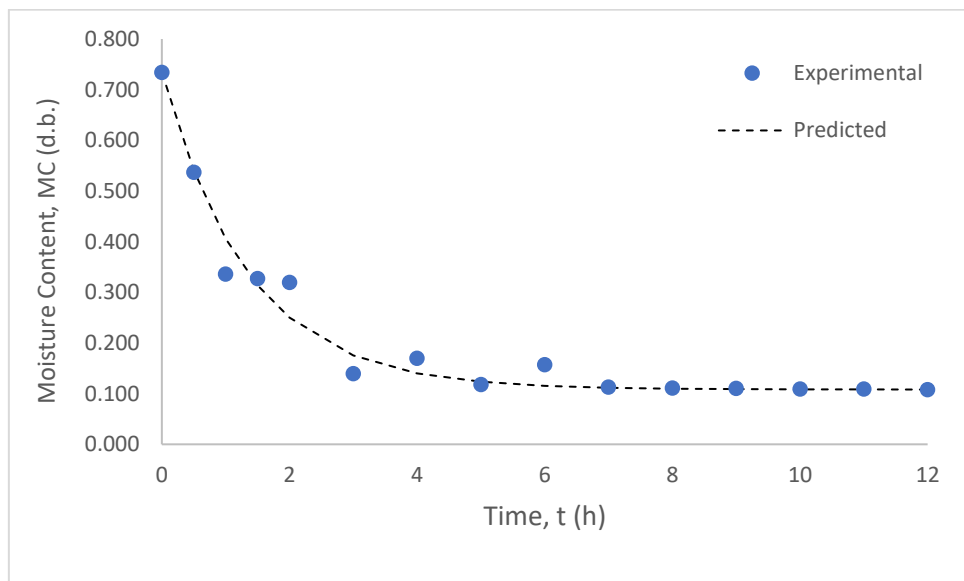


Figure 30: Predicted and experimental moisture content of sago starch using Lewis Newton model for intermittent drying

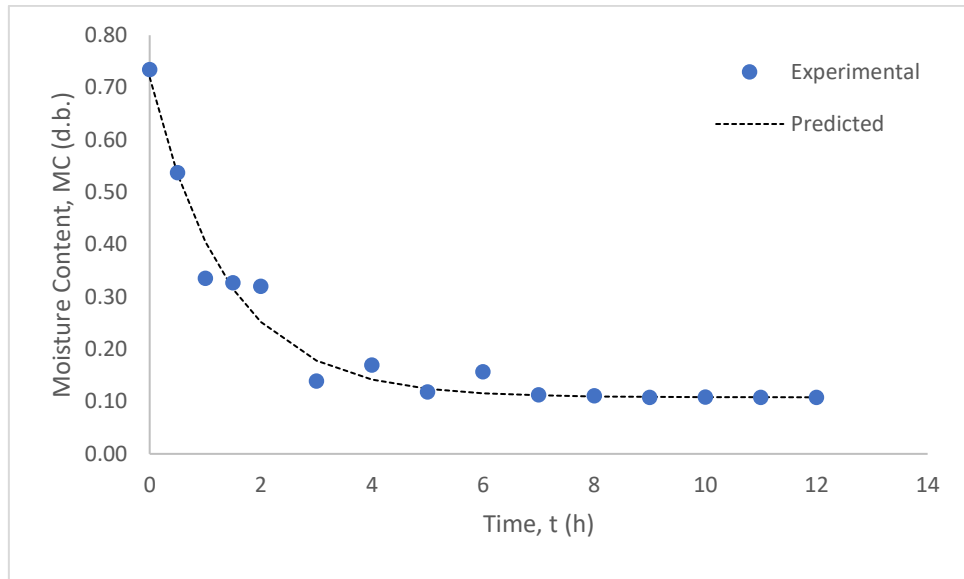


Figure 31: Predicted and experimental moisture content of sago starch using Henderson & Pabis model for intermittent drying

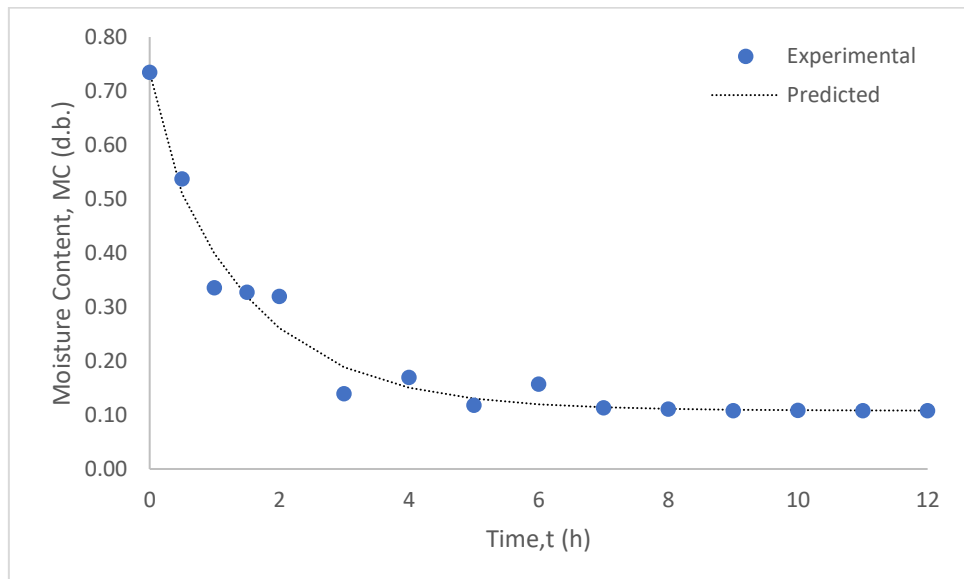


Figure 32: Predicted and experimental moisture content of sago starch using Modified Henderson & Pabis model for intermittent drying

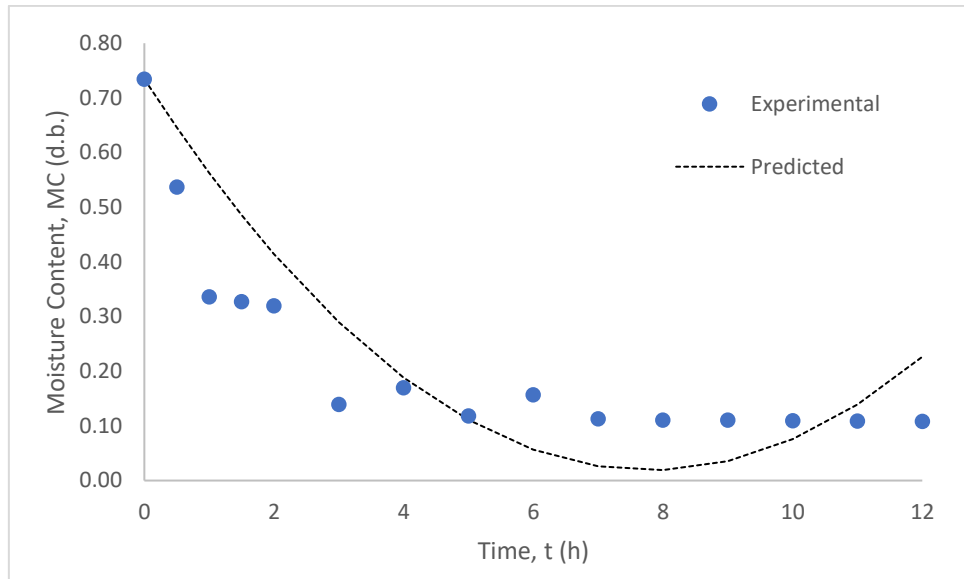


Figure 33: Predicted and experimental moisture content of sago starch using Wang & Singh model for intermittent drying

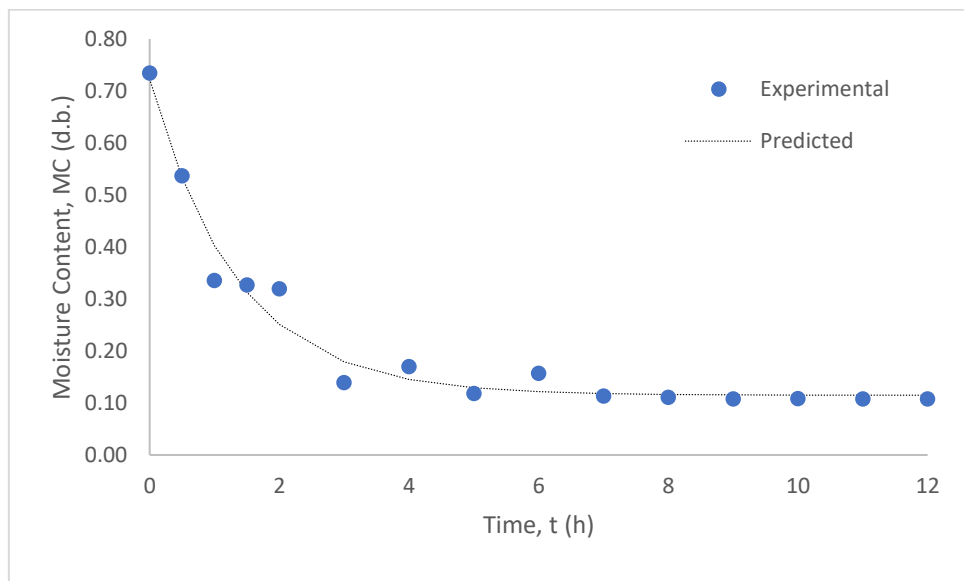


Figure 34: Predicted and experimental moisture content of sago starch using Logarithmic model for intermittent drying

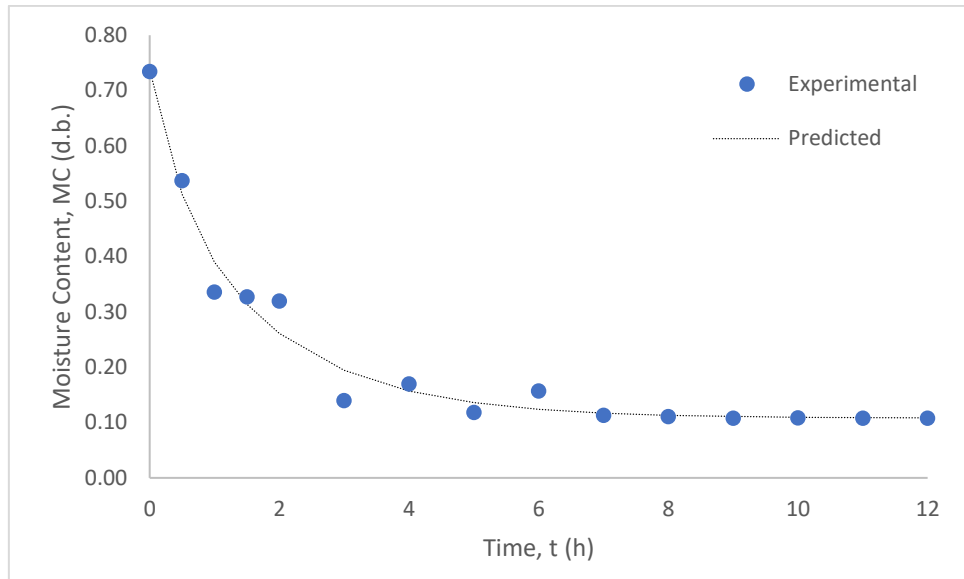


Figure 35: Predicted and experimental moisture content of sago starch using Two Term model for intermittent drying

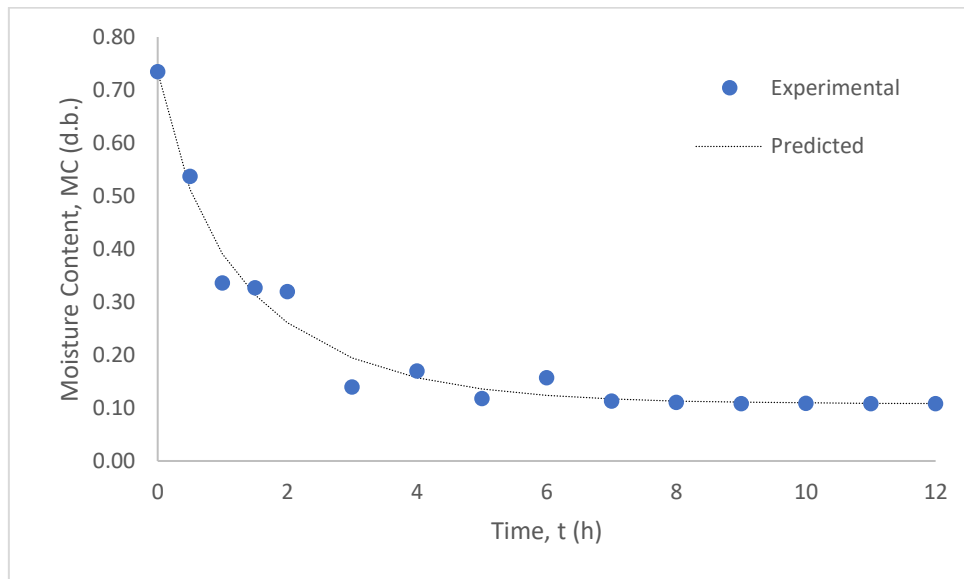


Figure 36: Predicted and experimental moisture content of sago starch using Approximation of Diffusion model for intermittent drying

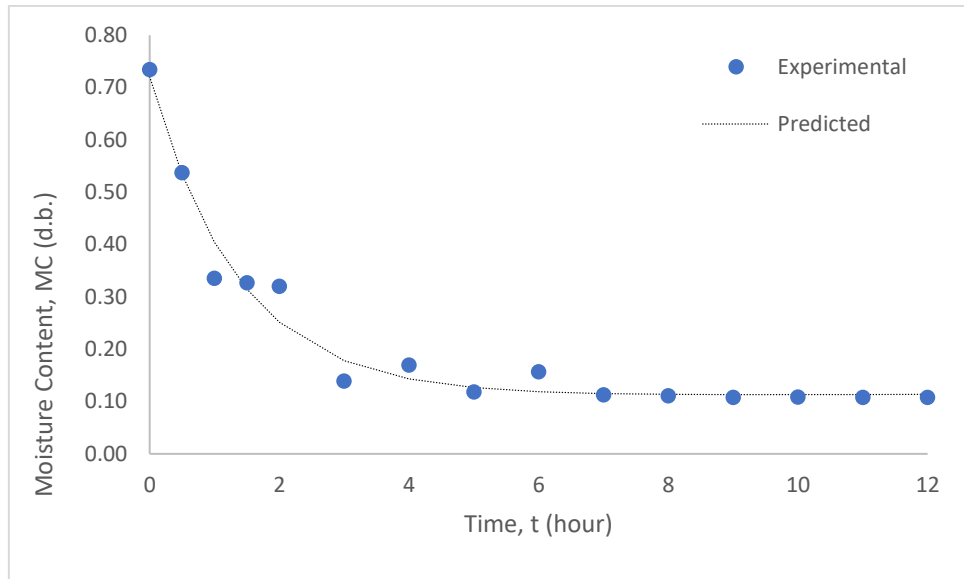


Figure 37: Predicted and experimental moisture content of sago starch using Midili Kucuk model for intermittent drying

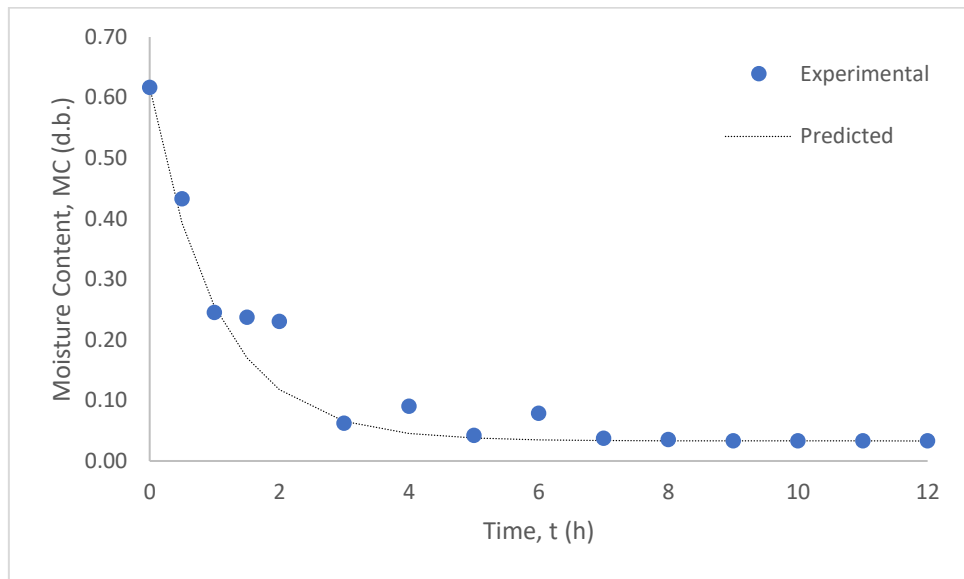


Figure 38: Predicted and experimental moisture content of sago starch using Verma model for intermittent drying

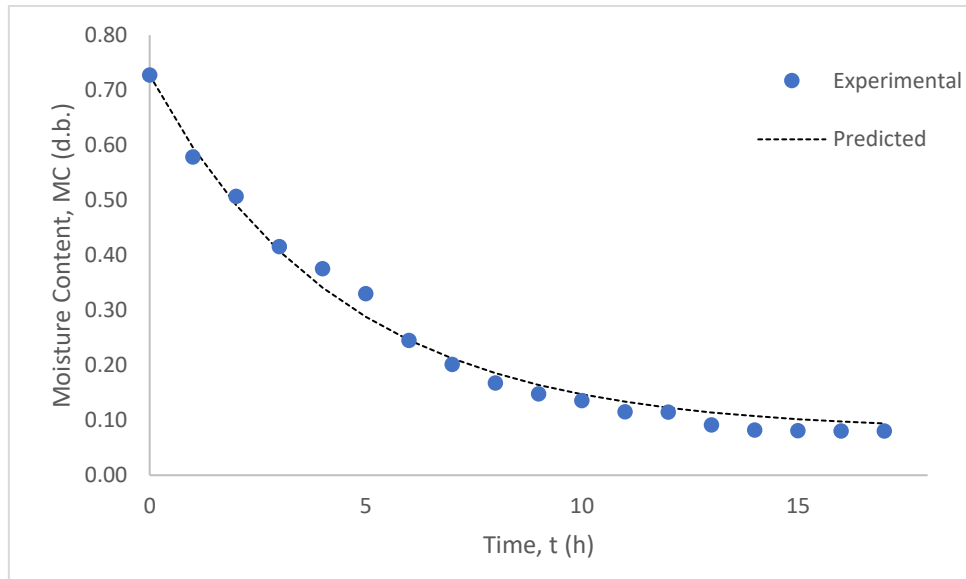


Figure 39: Predicted and experimental moisture content of sago starch using Lewis Newton model for sun drying

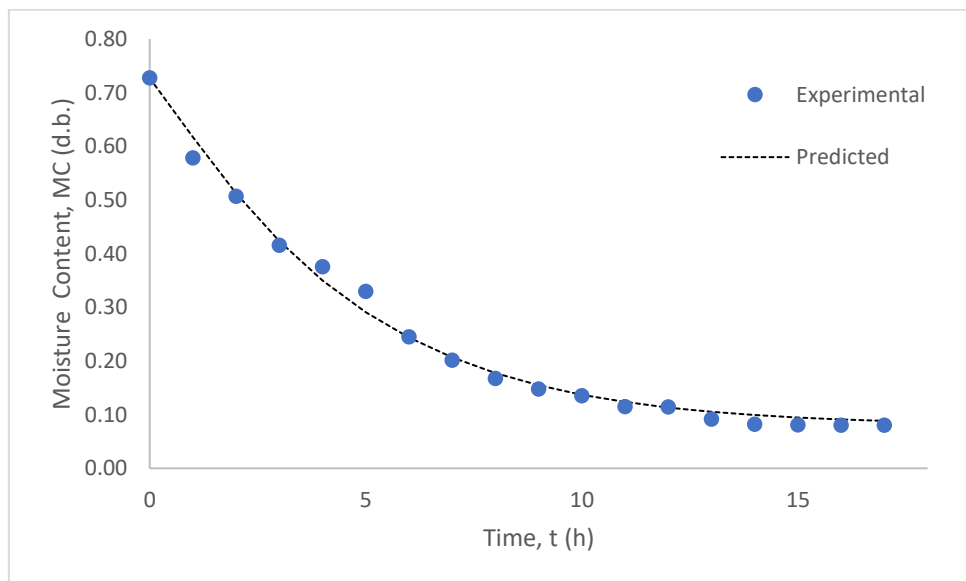


Figure 40: Predicted and experimental moisture content of sago starch using Page model for sun drying

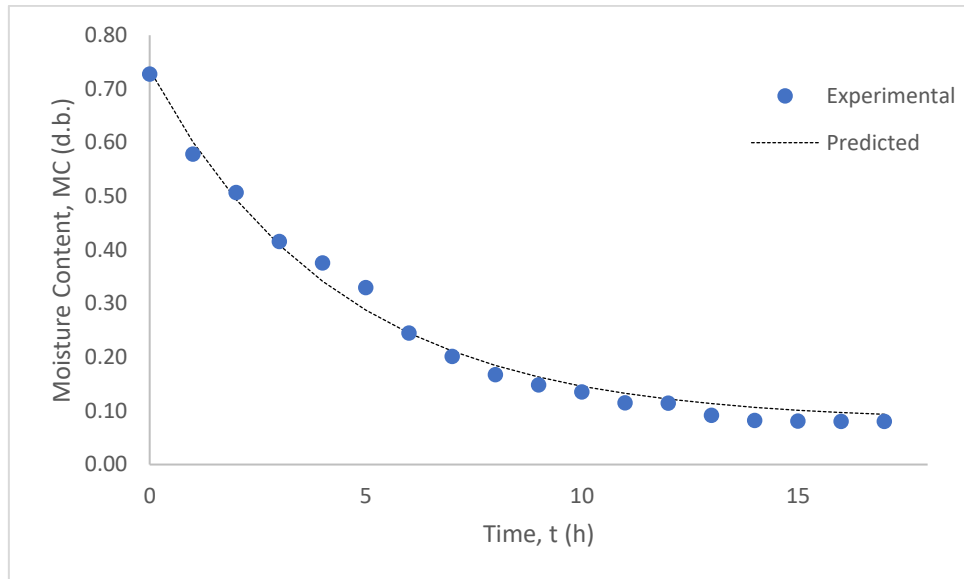


Figure 41: Predicted and experimental moisture content of sago starch using Henderson & Pabis model for sun drying

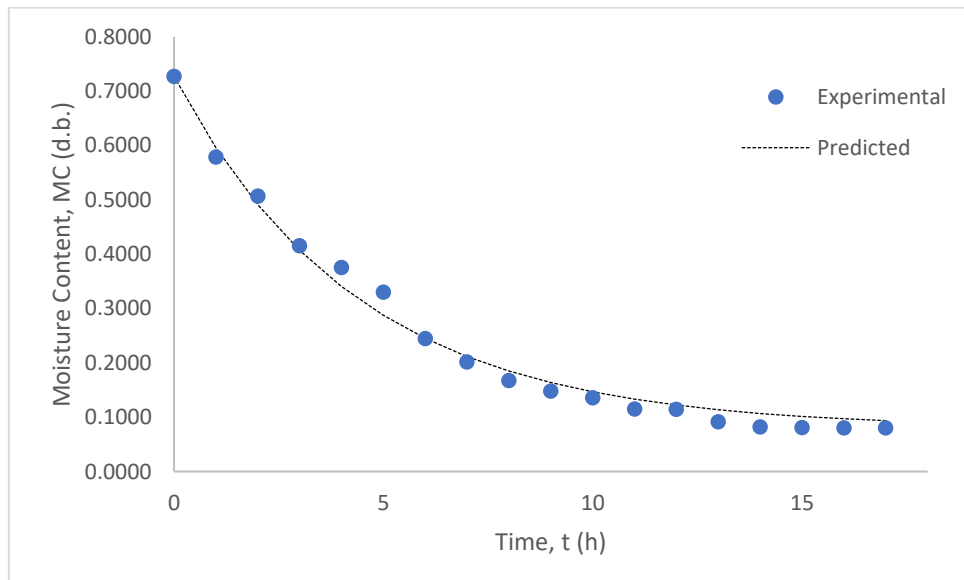


Figure 42: Predicted and experimental moisture content of sago starch using Modified Henderson & Pabis model for sun drying

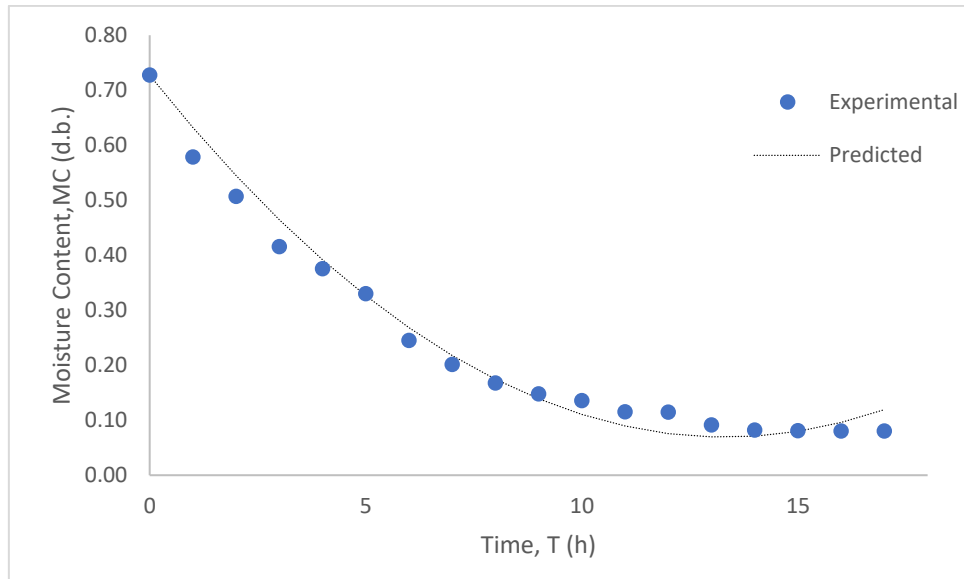


Figure 43: Predicted and experimental moisture content of sago starch using Wang & Singh model for sun drying

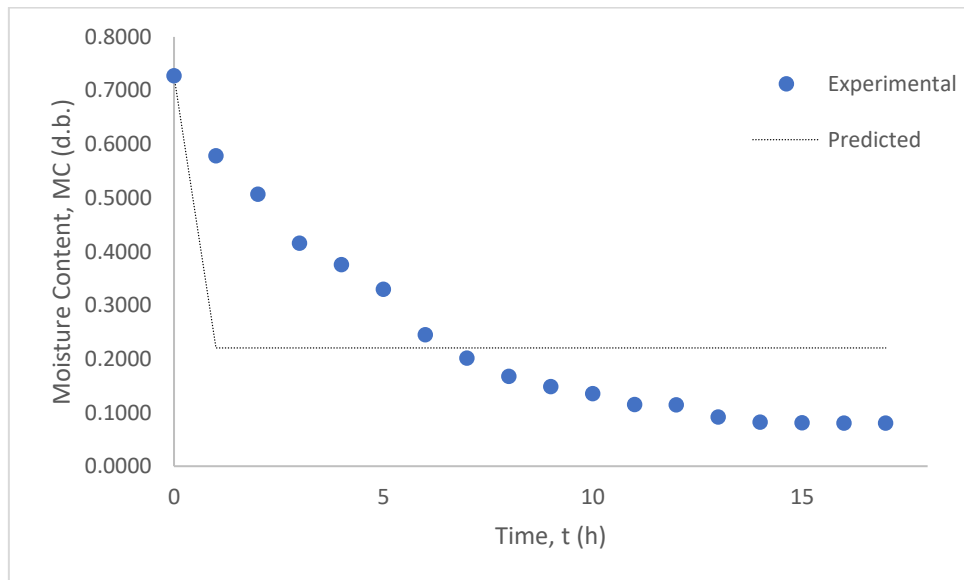


Figure 44: Predicted and experimental moisture content of sago starch using Logarithmic model for sun drying

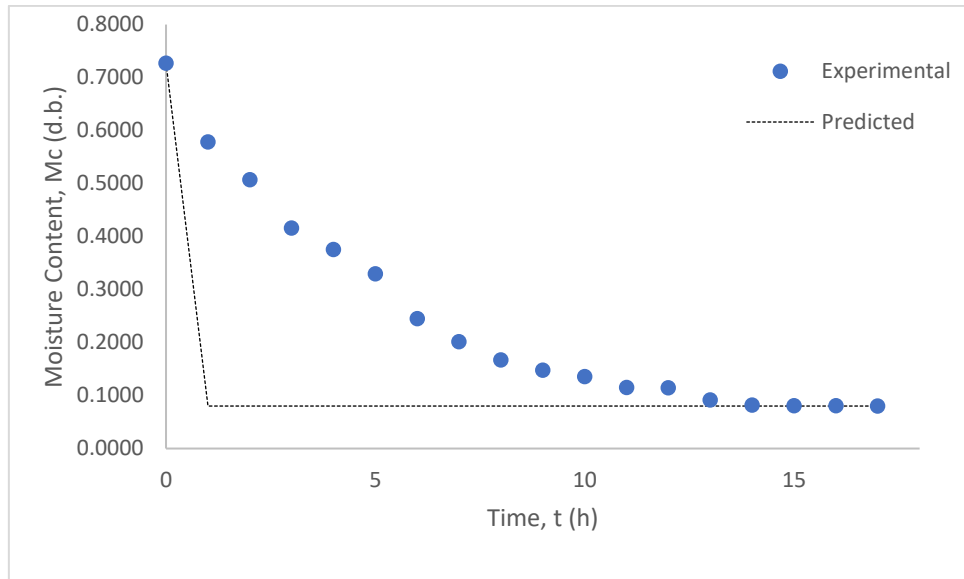


Figure 45: Predicted and experimental moisture content of sago starch using Two Term model for sun drying

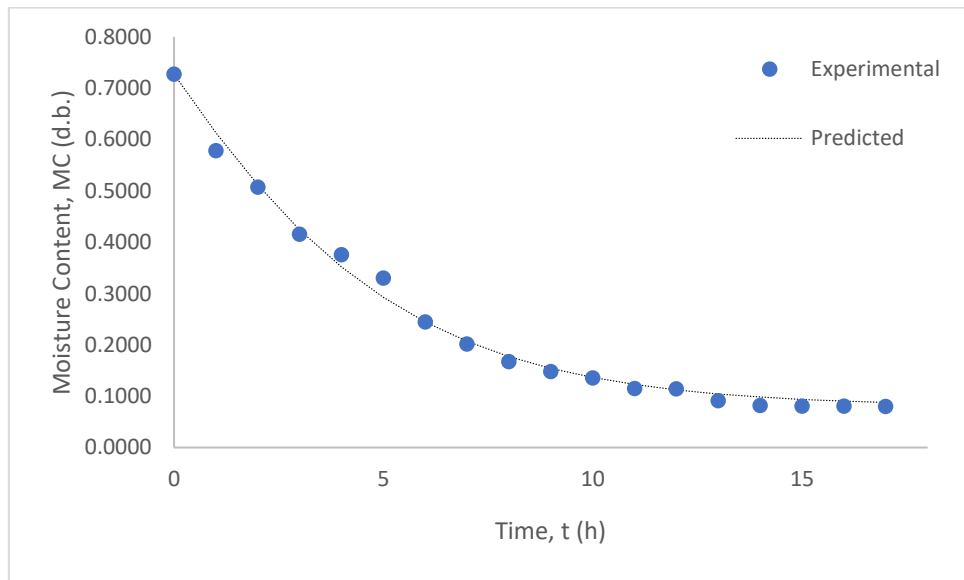


Figure 46: Predicted and experimental moisture content of sago starch using Approximation of Diffusion model for sun drying

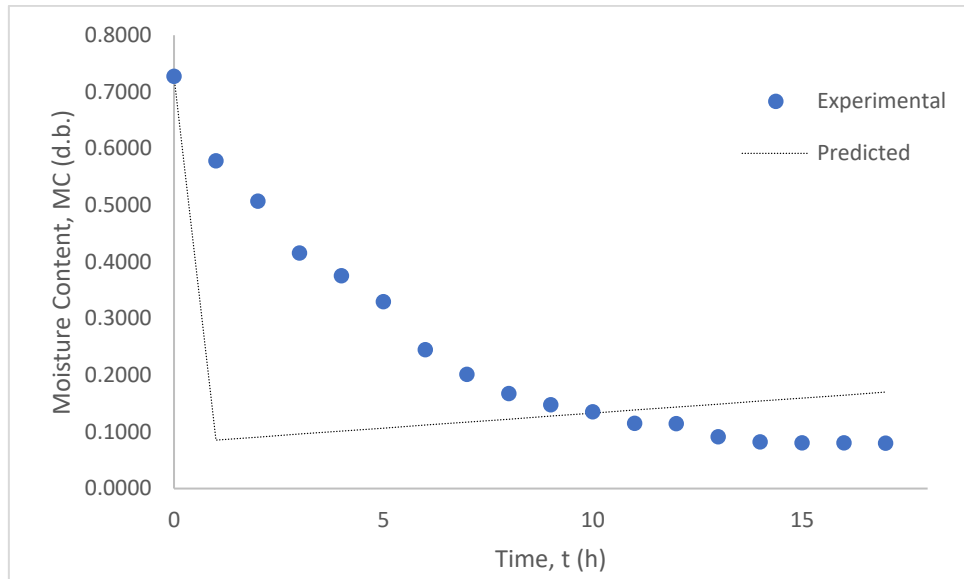


Figure 47: Predicted and experimental moisture content of sago starch using Midili Kucuk model for sun drying

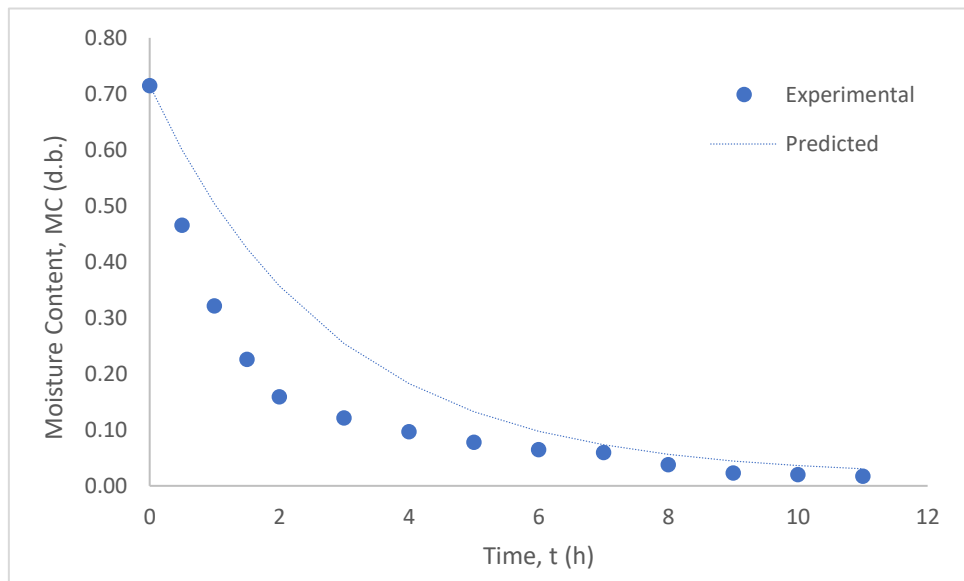


Figure 48: Predicted and experimental moisture content of sago starch using Lewis Newton model for freeze drying

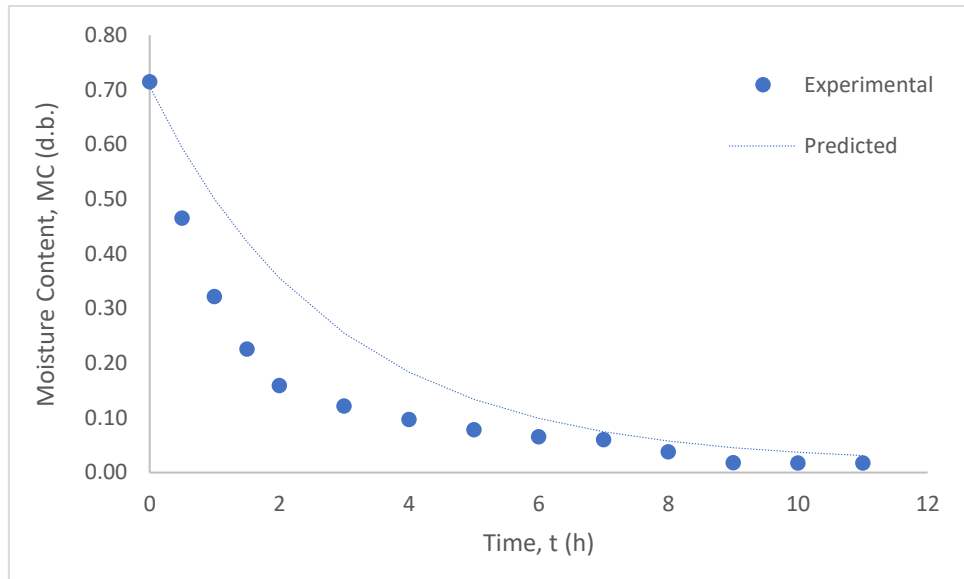


Figure 49: Predicted and experimental moisture content of sago starch using Henderson & Pabis model for freeze drying

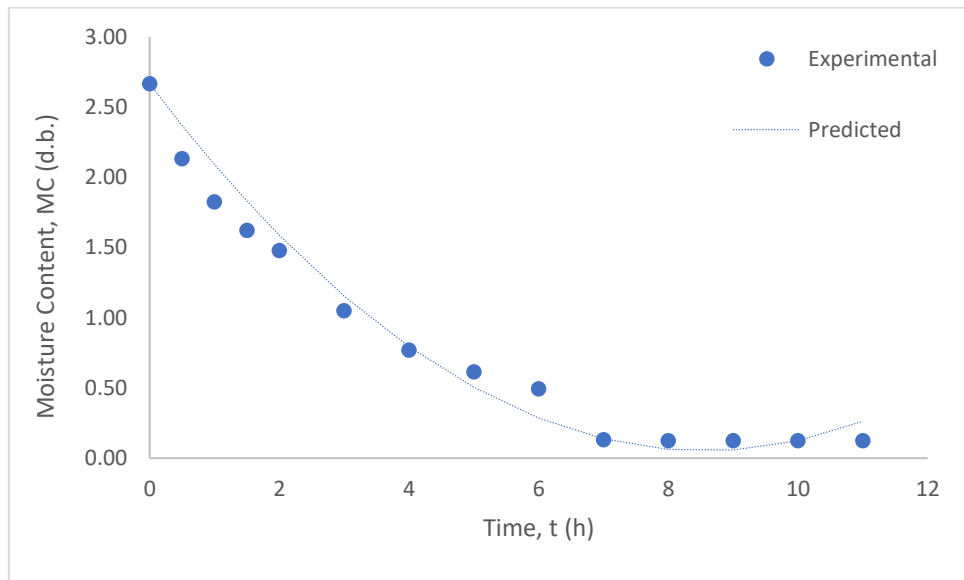


Figure 50: Predicted and experimental moisture content of sago starch using Wang & Singh model for freeze drying

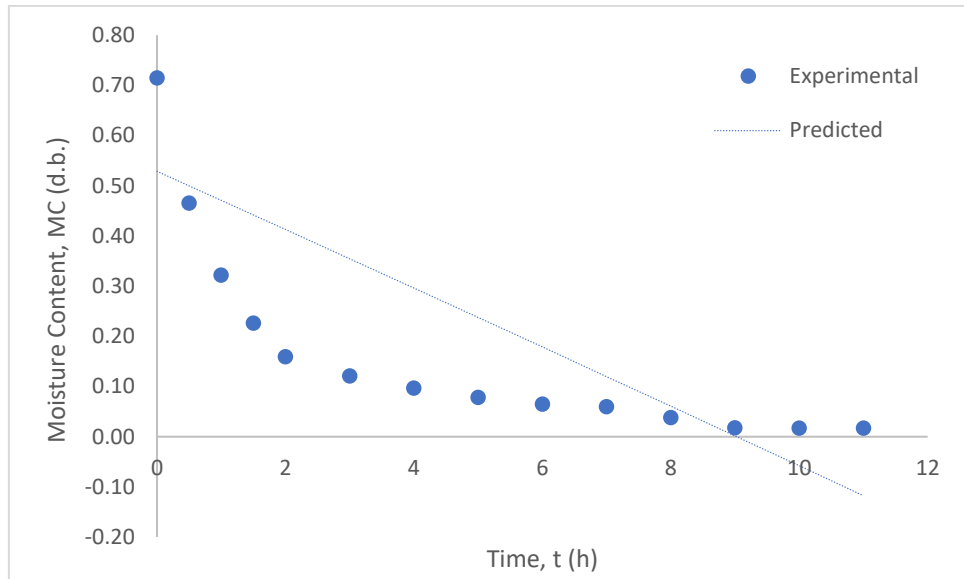


Figure 51: Predicted and experimental moisture content of sago starch using Logarithmic model for freeze drying

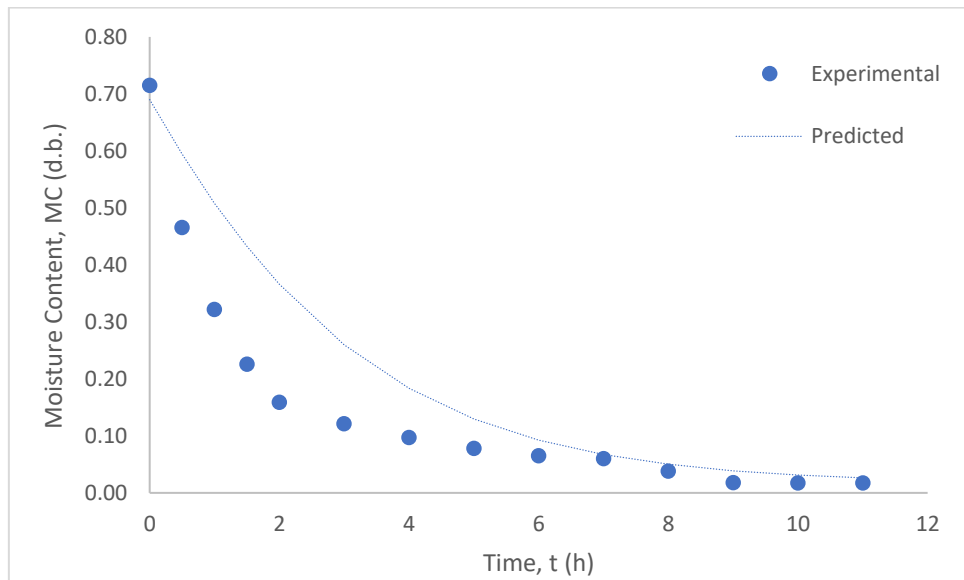


Figure 52: Predicted and experimental moisture content of sago starch using Two Term model for freeze drying

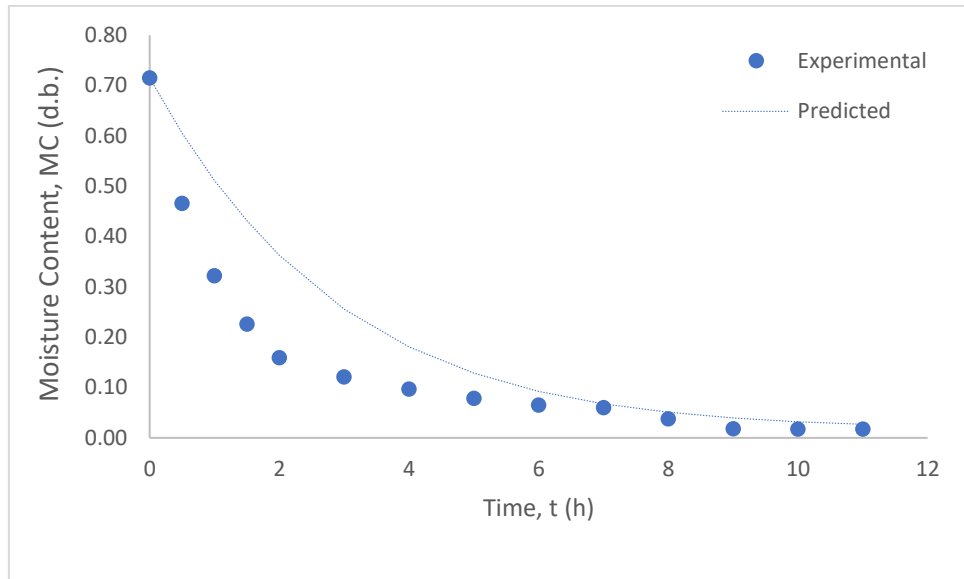


Figure 53: Predicted and experimental moisture content of sago starch using Approximation of Diffusion model for freeze drying

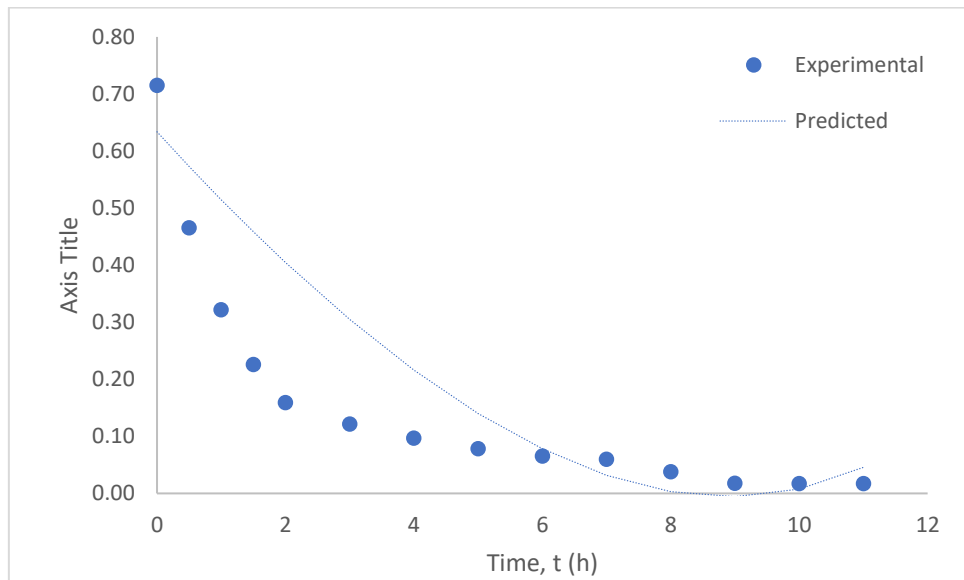


Figure 54: Predicted and experimental moisture content of sago starch using Midili Kucuk model for freeze drying

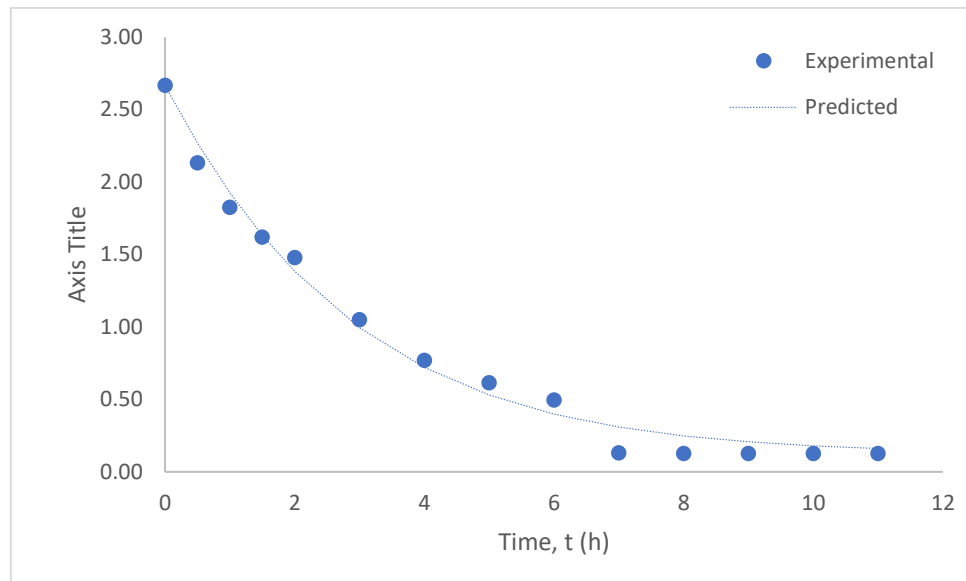


Figure 54: Predicted and experimental moisture content of sago starch using Verma model for freeze drying