

## Effect of microwave heating on corn flour and rice flour in water suspension

<sup>1</sup>\*Uthumporn, U., <sup>1</sup>Nadiah, N. I., <sup>1</sup>Koh, W. Y., <sup>2</sup>Zaibunnisa, A. H. and <sup>3</sup>Azwan, L.

<sup>1</sup>School of Industrial Technology, Universiti Sains Malaysia, 11800, Minden, Penang, Malaysia

<sup>2</sup>Faculty of Applied Sciences, Universiti Teknologi MARA, 40450 Shah Alam, Selangor

<sup>3</sup>School of Chemical Sciences & Food Technology, Faculty of Science and Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

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### Abstract

The effect of microwave heating and conventional heating towards the physicochemical and functional properties of corn and rice flour with 30% moisture content in water suspension at temperature of 50°C and 60°C were investigated. Conventional heat treatment was carried out at 50°C and 60°C respectively by direct heating the moisture treated flour. Microwave heating treatment was carried out by using domestic microwave oven at 50°C and 60°C respectively. The amylose content, particle size diameter, and gelatinization temperature are increasing in microwave and conventional heat treated corn and rice flour. Decreasing of pasting temperature, swelling power and solubility of all the heat-treated starches compared to control were detected. X-ray diffraction pattern of all control and heat treated corn and rice flour exhibit typical A-type pattern. Scanning Electron Microscopy (SEM) has revealed the heat treated corn and rice flour showed rougher surface, porous granules and rupture granules. There are no significance effect of temperature differences on corn and rice flour carried out at 50°C and 60°C. Evidently, microwave heating was effective to alter the physicochemical and functional properties of corn and rice flour.

### Keywords

Food biopolymer

Cereal flour

Microwave heating treatment

Amylose content

Particle size diameter

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### Introduction

Starch is accumulated in granules in endosperm, and the starch is deposited in layers with various amylose and amylopectin (Svihus *et al.*, 2005). Starch is one type of complex sugar which is polysaccharides built from a large number of glucose monomer. The two major glucose polymers are amylose and amylopectin which play an important role in functional properties of starch. Whereas, the minor constituents of starch accompanied by lipid, phosphorus, proteins and minerals which exert significant effect on functional properties.

Some example of cereal starch were corn and rice starch. Corn is the common name which used at United States for the cultivated member of the grass family (Gramineae) known to botanist as *Zea mays* L., but this crop is name as maize at the outside of United States (Eckhoff and Watson, 2009). Corn is believed to be a product of domestication in central Mexico beginning 5000-7000 years old (Eckhoff and Watson, 2009). Corn has been the staple food for countless generation for large group of people in Asia, North and South of America Native corn starch can use to thicken retail or institutional cook-and-serve products like gravies, sauces, pudding, and pie fillings (Mason, 2009). Moreover, corn starch can

be used in canned foods like soups, stews and chili which allowing the product to recover its consistency. Corn starch are widely use in food application in food field as corn starch consist of favorable functionality which able to enhance the food properties.

While, rice the major world food crop which known to botanist as *Oryza sativa* L., about 90% of the world's rice is produced and consumed in Asia (Mitchell, 2009). Rice starch is obtained by the alkaline steeping method with multi-stage purification, but commercial rice flour is produced by dry or wet milling process (Puncha-arnon and Uttapap, 2013). Rice is considered to be hypoallergenic, better digestibility, bland taste, whiteness and small granule size, those basic properties associated with rice has made it advantages over others (Mitchell, 2009). Rice starch has been used widely in food application such as binder in confectioneries, dusting agent in pharmaceuticals, binder in canned foods, crispness agent in extruded snacks and others. Commercial native rice starch is widely used in various applications such as cosmetic dusting powder, laundry stiffening agent, paper, soap and others. Rice starch has wider food application in food industry such as rice starch can act as thickener, binder, gelling, texturizing, stabilizing and others.

Microwave radiation is situated in the frequency

\*Corresponding author.

Email: [sapina@usm.my](mailto:sapina@usm.my)

Tel: +604-6532220; Fax: +604-6573678

interval of 300 MHz and 300 GHz within the electromagnetic spectrum. Generally microwave radiation in frequency range of 915 and 2450 MHz is for the industrial processing of foods. Whereas, domestic use microwave radiation is using the frequency of 2450 MHz. Microwave technology is playing an important role in the food industry as it had been used in variety applications such as tempering, thawing, drying, baking, rendering, pasteurization and sterilization. Microwave radiation is the non-ionizing energy capable of generating heat deep inside the penetrated medium by the “molecular friction” in an alternating magnetic field (Lewandowicz *et al.*, 1997). Traditionally, microwave heating of foods is being considered as a method to achieve fast and uniform thermal treatment of large sample volumes (Sakonidou *et al.*, 2003). The use of microwave radiation on the food is depends on the dielectric properties of the food product which are the microwave response characteristics of the food and the penetration depth (Fan *et al.*, 2013). The geometry, dielectric properties of the food and the characteristics of microwave sources and cavity substantially affect the development of the thermal profile of the samples (Sakonidou *et al.*, 2003). Currently, various studies had been carried out on the effect of microwave heating on variety of food substances in food industrial. Khraisheh *et al.* (2004) had made an investigation on the quality and structural changes in starchy foods during microwave and convective drying. This study had reported that there is a reduced in vitamin C destruction but a higher rehydration potential in the microwave dried samples.

Several researches had been carried out previously on the effect of microwave radiation on starch properties. For example, Lewandowicz *et al.* (2000) has reported that microwaved radiation of cereal starches had cause a shift in the gelatinization range to higher temperature and a drop in solubility and crystallinity. In addition, change of differences starch by the microwave radiation depends on both their crystal structure and amylose content. Modification of tuber starches by microwave radiation also has been carried out by Lewandowicz *et al.* (1997) which indicate that the microwave radiation was evidenced to affect the temperature and moisture content of starches with strong correlation between moisture content with rate of temperature rise. There was a rise in starch pasting temperature, drop in solubility and crystallinity. Palav and Seetharaman (2006) have investigated the effect of microwave heating on physicochemical changes in wheat starch model system. From this study, they have found that the

vibrational motion of polar water molecules and rapid increase in temperature cause the starch granules loss birefringence earlier than their gelatinization temperature.

Furthermore, the research on the impact of microwave treatment on the functionality of cereals and legumes has been carried out by Seema Ashraf *et al.* (2012). This investigation indicates that the microwave treated samples show greater functional properties than the untreated samples.

Therefore, this study was conducted to examine the effect of microwave radiation on corn and rice flour in order to compare with the effect of conventional heating treatment. The hypothesis of this study was microwave heating would induce changes in the functional, morphological and rheological properties of the corn flour and rice flour. This hypothesis was tested using rapid visco analyzer (RVA), differential scanning calorimeter (DSC), scanning electron microscopy (SEM), X-ray diffractometer, particle size analyzer, method of determine amylose content, swelling and solubility. Thus the objectives of this study were to study the effect of microwave heating compare to conventional heating on corn flour and rice flour using different temperatures (50°C and 60°C) and to determine the physicochemical and functional properties of both heating treatment flour at different temperature.

## Materials and Methods

### Materials

Corn flour and rice flour were obtained from commercial sources. Pure amylose and amylopectin from potato were purchased from Sigma Aldrich (Selangor, Malaysia).

### Microwave heating treatment

Slurry of corn flour and rice flour with concentration (30% w/v) was prepared by mixing 30 g of flour to 100 mL of distilled water. Slurry was stirred by using glass ball uniformly and placed in the microwave oven (Panasonic, Model: NN-GD570S, 2450 MHz) for heating to reach temperature of 50°C and 60°C. Temperature of the flours was monitored by using thermometer. Temperature of 50°C and 60°C was choosing for heating treatment based on the result done in preliminary test which below gelatinization temperature determined by Rapid Visco Analyzer (RVA). Then, samples were recovered by filtration using the Whatman no.1 filter paper. The starch cake was dried in a convention oven at 40°C for 48 hours as the samples achieved desirable moisture content (9-10%) and stored the samples at room temperature

in a sealed polyethylene bag for further analysis.

#### *Conventional heating treatment*

Slurry of flours were heated by using hot plate to reach temperature of 50°C and 60°C, heating and stirring with magnetic stirrer bar was carried out simultaneously to get a fully suspended solution. Untreated corn flour and rice flour was adjusted to 30% moisture content and used as a control. Same as microwave heating treatment, samples were recovered by filtration using the Whatman no.1 filter paper. The starch cake was dried in a convention oven at 40°C for 48 hours as the samples achieved desirable moisture content (9-10%) and stored the samples at room temperature in a sealed polyethylene bag for further analysis.

#### *Determination of amylose content*

Amylose content of each samples was determined through the UV-Vis spectrophotometer based on the method describe by McGrance *et al.* (1998) with modification. Flour sample (0.1 g, dry basis) was dissolved in 2 ml of dimethyl sulfoxide (DMSO) and heated at 90°C on a hot plate for 15 min with stirring using magnetic stirrer bar. 25 ml of deionized water was added to dilute the mixer and 1 ml of aliquot was diluted to 50 ml deionized water. 5 ml of iodine (I<sub>2</sub>) in potassium iodide (KI) was added to the solution. The absorbance of solution was developed after 15 min and the reading was obtained at 600 nm through UV-Vis spectrophotometer. Pure potato amylose and amylopectin were used as the standards.

#### *Determination of particle size distribution*

Granule size distributions of each sample was determined by using a low angle laser light scattering (Mastersizer S, Malven Instruments Malvern, UK). Samples must keep in the oven overnight to remove the moisture content in order to prevent the clumping of the samples before carry out the analysis.

#### *Determination of swelling and solubility*

Swelling power and solubility were analyzed based on the method of Schoch (1964) with modification. Flour samples (0.1 g, dry basis) were weighed in a 50 ml graduated centrifuge tube and added with distilled water. The suspension was heated in a shaking water bath at a desired temperature 95°C for 30 min. The mixture was cooled to room temperature and centrifuged (4000 × g, 15 min). After the centrifugation, the residue was weighed for the calculation of swelling power of starch. The supernatant after centrifugation was dried to a constant weight overnight in an oven at 110°C. The

solubility of starch can be calculated by using weight of dried supernatant.

$$\text{Swelling power (g/g)} = \frac{[\text{weight of residuc after centrifuged, g}]}{[\text{weight of sample in dry basis, g}]}$$

$$\text{Solubility (\%)} = \frac{[\text{weight of dried supernatant, g}]}{[\text{weight of sample in dry basis, g}]}$$

#### *Determination of pasting properties*

Pasting properties of flour samples were determined by using Rapid Visco Analyzer (RVA, Model 3D-plus, Newport Scientific, Narrabeen, Australia). Flour sample (3 g, 10% moisture contents) was weighed and mixed with 25 ml of distilled water and stirred in RVA test aluminium canister. Temperature was held at 50°C for 1 min and then raised to 95°C in 3.75 min, held for 2.5 min, cooled to 50°C in 3.75 min and held for 5 min. The paddle speed was set at 960 rpm for the first 10 seconds, and then reduced to 160 rpm throughout entire analysis. The units of viscosity were expressed as cP.

#### *Determination of thermal properties*

Thermal properties, the gelatinization properties of flour sample was analyzed by using Differential Scanning Calorimeter (Model DSC 2910, Du Pont Instrument, Delaware, USA) based on procedure described by Lewandowicz *et al.* (2000). Approximate 2 mg of the sample was weighed directly in aluminium pan and water added to a starch/water ratio of 1:3 and hermetically sealed. An empty pan was used as a reference. The pan was left for 2 hours at room temperature to equilibrate the sample and then heated from 30 to 130°C at the rate of 10°C/min.

#### *Scanning electron microscopy*

The microstructure of the starch granules of each sample was observed by using a field emission scanning electron microscope (FESEM Leo Supra 50VP, Carl-Ziess SMT, Oberkochen, Germany). The accelerating voltage of 5 kV was using in the analysis. Flour sample was mounted on the aluminium specimen stubs with double adhesive tape and sputter-coated with layer of gold by using a Sputter Coater [Polaron (Fisions) SC515, VG Microtech, Sussex, UK].

#### *X-ray diffraction*

The crystallinity patterns of starch granule for each sample were examined under X-ray diffractometer (Diffractometer D5000, SIEMENS, Karlsruhe, Germany). X-ray diffractometer was using follow conditions which is X-ray tube CuK $\alpha$  with a nickel filter, voltage 40 kV, current 25 mA, and scanning through 2 $\theta$  = 5° to 40°.

### Statistical analysis

All analyses of flour characteristics and properties were carried out in three replications. All data was reported as mean values and standard deviations. The experimental data were subjected to one-way analysis of variances (ANOVA) by using the SPSS version 16.0 (SPSS Inc., USA). Duncan's test was conducted to examine significant differences among experimental mean values ( $P < 0.05$ ).

## Results and Discussion

### Amylose content of control and treated starches

The amylose content of control, conventional heating treated and microwave heating treated corn and rice flour are listed in Table 1. The results show that amylose content for corn control is  $22.37 \pm 0.44\%$  while the amylose content for rice control is  $19.67 \pm 0.36\%$ . McGrance *et al.* (1998) reported that the amylose contents of commercial corn starches which determined by measurement of the iodine complex at 600 nm are 23.1% to 24.7%. Previous study by Biliaderis *et al.* (1980) on starch gelatinization phenomena has been shown that amylose in commercial corn starches was 22.6%. Whereas amylose content of rice control is  $19.67 \pm 0.36\%$  which consistent with the finding by (Mitchell, 2009) show that common rice starches contain around 20% of amylose. Extend of the increase of amylose content followed the order: control < conventional treated < microwave treated. Amylose content of heating treated flour had increased significantly compared to control. This might due to the heating cause the degradation of amylose and amylopectin in starch granules, thus, increase the amylose content. Different temperature of microwave and conventional heating which is at 50°C and 60°C for this study has shown an insignificance effect on amylose content of flour. Except the microwave treated corn flour at 60°C had shown significant differences compare with the microwave 50°C treatment. Highest amount of amylose content was detected on microwave heated sample; the results indicated that amylose content of cereal flour was affected more by the microwave heat treatment vis-à-vis conventional heating treatment. Weakening of the interaction between amylose and amylopectin, and improvement of dispersion and separation after microwave treatment caused starch chains more readily react with the iodine thus increasing the amylose content of flour (Lewandowicz *et al.*, 2000; Zhao *et al.*, 2007). Moreover, microwave power had destroyed the starch structure thus increase the amylose content had been proven, which previously

reported by Zhang *et al.* (2009).

### Particle size distribution of control and treated starches

The particle size distribution of control, conventional heating treated and microwave heating treated corn and rice flour are presented in Table 1. Discrepancies in mean diameter of corn flour and rice flour shown in the results might due to the differences in morphological structure of starch granules. A drastic increment can be observed from the changes of mean diameter of heat treated flour compare with control. The results had shown that conventional heating and microwave heating treatment have greater effect on the particle size distribution of corn and rice flour. Higher values of means diameter was detected for microwave irradiated sample indicated that microwave heating have more effect on the particle size of cereal flour. Fan *et al.* (2012) reported that there is an increase of granule size of microwave and conventional heated starch which observed from the temperature-granule size profile of starch dispersions during microwave heating and rapid conventional heating. The result is correlated with the result of SEM as heat treated starch show an increment in the particle size compare with the control. From the result shown in Table 1, a greater change is exhibited in the means diameter of starch for both heating between 50°C and 60°C. This finding indicated that temperature difference does affect both particles diameter of conventional heated and microwave heated corn and rice flour. This phenomenon is due to the irreversible swelling of the starch granules to a certain degree resulting a marked increasing in the diameter of the granules.

### Swelling power and solubility of control and treated starches

Swelling power and solubility play an important role in starch as both functional properties to provide the crystalline nature of starch granules. Difference of swelling power and solubility was attributed to different of amylose content and weak internal structure. The swelling power and solubility of control and heating treated corn and rice flour are summarized in Table 1.

Swelling power of the corn control is  $13.97 \pm 0.38$  g/g which accordance with the swelling power varying from 13.7 g/g to 20.7 g/g from different corn varieties previously reported by Sandhu & Singh (2007). Solubility of corn flour control shown in the table is  $9.57 \pm 0.33\%$ . Ayucitra (2012) had observed the swelling power and solubility of native corn starches are 15.5 g/g and 8.50% respectively. Rice

Table 1. The amylose content of control, particle size distribution, swelling power and solubility of control, conventional heating treated and microwave heating treated corn and rice flour

Sample	Amylose Content (%)	Particle diameters (µm)	Swelling power (g/g)	Solubility (%)	
Corn	Control	199.40 <sup>f</sup> ±1.05	13.97 <sup>d</sup> ±0.38	9.57 <sup>g</sup> ±0.33	22.37 <sup>cd</sup> ±0.44
	CV 50°C	23.58 <sup>g</sup> ±0.36	209.60 <sup>h</sup> ±1.77	13.58 <sup>cd</sup> ±0.67	9.35 <sup>g</sup> ±0.31
	CV 60°C	23.65 <sup>g</sup> ±0.87	214.13 <sup>h</sup> ±1.63	13.48 <sup>cd</sup> ±0.66	8.50 <sup>f</sup> ±0.60
	MW 50°C	25.02 <sup>g</sup> ±0.35	216.58 <sup>h</sup> ±1.41	12.96 <sup>cd</sup> ±0.11	7.65 <sup>g</sup> ±0.44
	MW 60°C	26.63 <sup>g</sup> ±0.44	321.12 <sup>i</sup> ±1.57	12.71 <sup>cd</sup> ±0.50	7.54 <sup>g</sup> ±0.44
Rice	Control	65.75 <sup>g</sup> ±1.77	13.26 <sup>g</sup> ±0.54	5.49 <sup>g</sup> ±0.37	19.67 <sup>g</sup> ±0.36
	CV 50°C	21.18 <sup>g</sup> ±0.88	76.12 <sup>h</sup> ±3.89	12.77 <sup>cd</sup> ±0.71	4.98 <sup>g</sup> ±0.40
	CV 60°C	21.91 <sup>g</sup> ±0.17	115.61 <sup>h</sup> ±1.64	12.57 <sup>cd</sup> ±0.30	4.41 <sup>g</sup> ±0.39
	MW 50°C	23.18 <sup>g</sup> ±0.19	120.61 <sup>h</sup> ±2.22	11.59 <sup>g</sup> ±0.22	2.92 <sup>g</sup> ±0.32
	MW 60°C	23.93 <sup>g</sup> ±0.32	183.99 <sup>h</sup> ±3.13	11.57 <sup>g</sup> ±0.21	2.03 <sup>g</sup> ±0.10

Valued expressed as mean ± standard deviations of three independent experiments (n=3). Distinct letters represent statistical differences for the mean values at (P>0.05)

Table 2. Pasting properties of control, conventional heating treated and microwave heating treated corn and rice flour

Sample	Pasting Temperature (°C)	Viscosity (cP)				
		Setback	Peak Viscosity	Final Viscosity	Breakdown	
Corn	Control	80.12 <sup>g</sup> ±0.46	1106.33 <sup>g</sup> ±4.00	3057.67 <sup>g</sup> ±35.22	3285.67 <sup>g</sup> ±28.85	878.33 <sup>g</sup> ±44.00
	CV 50C	83.83 <sup>g</sup> ±0.03	1084.33 <sup>g</sup> ±2.83	2901.67 <sup>g</sup> ±23.63	3198.67 <sup>g</sup> ±12.66	787.33 <sup>g</sup> ±17.21
	CV 60C	84.13 <sup>g</sup> ±0.49	1107.33 <sup>g</sup> ±5.11	2877.00 <sup>g</sup> ±63.59	3220.33 <sup>g</sup> ±87.23	764.00 <sup>g</sup> ±47.28
	MV 50C	84.47 <sup>g</sup> ±0.40	1083.03 <sup>g</sup> ±9.85	2883.00 <sup>g</sup> ±19.97	3192.00 <sup>g</sup> ±9.17	774.00 <sup>g</sup> ±37.64
	MV 60C	85.83 <sup>g</sup> ±0.45	967.67 <sup>g</sup> ±1.91	2784.33 <sup>g</sup> ±36.83	3132.67 <sup>g</sup> ±34.20	602.67 <sup>g</sup> ±23.71
Rice	Control	82.07 <sup>g</sup> ±0.45	2130.73 <sup>g</sup> ±2.09	2909.33 <sup>g</sup> ±165.78	4245.67 <sup>g</sup> ±398.99	797.67 <sup>g</sup> ±29.94
	CV 50C	82.23 <sup>g</sup> ±0.03	2177.73 <sup>g</sup> ±3.72	2862.67 <sup>g</sup> ±81.43	4447.00 <sup>g</sup> ±259.65	713.33 <sup>g</sup> ±40.70
	CV 60C	82.87 <sup>g</sup> ±0.45	2128.33 <sup>g</sup> ±2.32	2850.33 <sup>g</sup> ±116.51	4239.67 <sup>g</sup> ±403.96	739.00 <sup>g</sup> ±69.20
	MV 50C	82.90 <sup>g</sup> ±0.44	2103.33 <sup>g</sup> ±1.54	2830.67 <sup>g</sup> ±30.86	4225.33 <sup>g</sup> ±173.66	708.67 <sup>g</sup> ±26.02
	MV 60C	83.68 <sup>g</sup> ±0.46	2127.73 <sup>g</sup> ±1.45	2827.00 <sup>g</sup> ±40.84	4262.00 <sup>g</sup> ±142.76	659.33 <sup>g</sup> ±59.28

Valued expressed as mean ± standard deviations of three independent experiments (n=3). Distinct letters represent statistical differences for the mean values at (P>0.05)

control has shown swelling power and solubility which is 13.26 ± 0.54 g/g and 5.49 ± 0.37% respectively. Yu *et al.* (2012) has observed that rice starch and rice flour from different rice cultivars has the swelling power is ranged from 12.11 to 15.98 g/g and solubility ranged from 6.28 to 7.06%. A marked decrease in swelling power and solubility of both heating treatments can be observed from the results. Control starch has shown a higher value of swelling power and solubility compare with heat-treated starch due to the structure of untreated starch granules are more rigid with less swelling and not easily be broken. Decreasing of swelling power and solubility of microwave irradiated starches is in accordance with previous study by Lewandowicz *et al.* (2000) who mentioned that microwave irradiation was

evidences to reduce the swelling power and solubility of cereal starches. According to Luo *et al.* (2006), both swelling power and solubility of maize starch were decreased following microwave radiation. The reduction in swelling power and solubility appears to be quite similar for both conventional heating and microwave heating. Lowest amount of swelling power and solubility were detected by microwave heated samples indicated microwave heating has made a more significant effect on the swelling and solubility characteristics of starches. Lewandowicz *et al.* (2000) stated that microwave treatment has caused some structural changes which decreased the swelling characteristics and make the solubilization of corn starches in water more difficult.

Whereas, temperature differences of both heating

treatment has shown an insignificant differences on the swelling and solubility characteristics of cereal flour. This indicated that 50°C and 60°C heating treatment may not bring much change on the structure of starch granules. According to Tester and Morrison (1990), amylose often correlated with lipid in cereal starches which amylose-lipid complexes likely to be formed inhibiting the swelling of cereal starches. Thus, formation of amylose-lipid complexes might cause the reduction of swelling power. Previous studies on effect of microwave irradiation on potato and tapioca starches has shown that starch-starch bonds are stronger in the irradiated starches which reported by Lewandowicz *et al.* (1997). Microwave heating had caused some intra-granular molecular rearrangement which lead to lesser accessibility to amorphous area, limiting the swelling and solubilization of starch granules (González and Pérez, 2002). Certain arrangement of crystalline regions within starch granules which become more randomly distributed in starch granules might causing the reduction of swelling power in starch (Luo *et al.*, 2006). Yet, Lewandowicz *et al.* (2000) found that the significant reduction of swelling power of cereal starches may due to changes in amorphous amylose part of starch granule rather than crystallization occurs during microwave treatment.

#### *Pasting properties of control and treated starches*

The pasting properties of control, conventional heated and microwave heated corn and rice flour analyzed by RVA are presented in Table 2. Stevenson *et al.* (2005) had observed that irradiated corn starch have high pasting temperature and lower peak, final, breakdown and setback viscosity than the native corn starch. The finding from Stevenson *et al.* (2005) is similar with the result shown in Table 2.

Conventional heating treated corn and rice flour show an increase trend on pasting temperature compare to the control. Puncta-arnon and Uttapap (2013) had observed that pasting temperature of heat-moisture treated rice flour and rice starch with 20%, 25% and 30% is increasing compared with the untreated sample. The same pattern of the results also had been shown by Jiranuntakul *et al.* (2001) on rice and corn starches. A greater change is exhibit on pasting temperature of microwave irradiated corn and rice flour at 60°C. Microwave irradiated flour exerts more effect on the starch vis-à-vis conventional heating as corn and rice flour show the marked highest of pasting temperature. According to Stevenson *et al.* (2005), pasting temperature of irradiated starch with 25-40% moisture is higher than native corn starch. Rise in pasting temperature as a

result of irradiation on wheat and corn starch had been studied by Lewandowicz *et al.* (2000). Setback is used to describe the increase in viscosity which occurs on cooling a pasted starch that associated with retro gradation phenomena (Fisher and Thompson, 1997; Anderson *et al.*, 2006). The results have shown a decreasing of setback viscosity of heat treated starch compare to the control. The reducing of setback viscosity may due to the lower tendency for re-aggregation in starch granule after conventional heating and microwave heating treatment.

The decreasing of peak viscosity correlated with reducing of swelling ability and water binding capability of starch granules. A decrease trend can be observed from the peak viscosity of heat treated corn and rice flour. Insignificant differences exhibit in peak viscosity of heat treated rice flour compare with rice control. However, heat treated corn flour exhibit significant lower peak viscosity than corn control. The thermal degradation of amylopectin and amylose granules was causing the reduction of peak viscosity. Breakdown viscosity of microwave irradiated and conventional heated of corn and rice flour show lower value than the control. A high breakdown value is associated with high peak viscosities which also correlated with the swelling of starch (Ragaee and Abdel-Aal, 2006). Decreasing of breakdown viscosity reflected starch structure more stable. Decrease in viscosity can be observed from the results is in accordance with previous studies on effect of microwave radiation on cereal starches had shown a drop in viscosity on irradiated tuber and cereal starches (Lewandowicz *et al.*, 1997; Lewandowicz *et al.*, 2000; Stevenson *et al.* 2005). The changes in pasting properties on conventional heated and microwave heated corn and rice flour are due to the structural rearrangement and starch-chain association during heat treatment (Puncta-arnon and Uttapap, 2013). Moreover, phase separation between amylose and amylopectin, compaction of granular matter by vapor pressure force, and chemical bonding or interactions may be the factors which affect the strength of starch thus affect the pasting properties (Watcharactewinkul *et al.*, 2009).

#### *Thermal Properties of control and treated starches*

Starch gelatinization is a process which involved granules swelling due to absorption of moisture in amorphous regions, leaching of small amylose, loss crystalline order, leaching of large amylopectin from the granule and finally disintegration of starch granules (Sakonidou *et al.*, 2003; Fan *et al.*, 2012). Thermal properties of control, conventional heating treated and microwave heating treated corn and rice

Table 3. Thermal properties of control, conventional heating treated and microwave heating treated corn and rice flour

Sample	Gelatinization				
	To (°C)	Tp (°C)	Tc (°C)	ΔH (J/g)	
Corn	Control	61.60 <sup>a</sup> ±2.09	62.76 <sup>a</sup> ±2.24	63.68 <sup>a</sup> ±2.21	11.84 <sup>a</sup> ±0.50
	CV50°C	62.07 <sup>a</sup> ±1.36	63.63 <sup>a</sup> ±1.82	64.36 <sup>ab</sup> ±1.36	11.26 <sup>ab</sup> ±0.16
	CV60°C	62.29 <sup>a</sup> ±0.94	64.05 <sup>a</sup> ±1.24	64.96 <sup>ab</sup> ±0.98	10.61 <sup>a</sup> ±0.37
	MV50°C	62.93 <sup>a</sup> ±0.70	64.32 <sup>a</sup> ±0.93	65.27 <sup>ab</sup> ±0.54	8.72 <sup>b</sup> ±0.37
	MV60°C	63.54 <sup>a</sup> ±1.92	65.07 <sup>a</sup> ±1.29	66.23 <sup>b</sup> ±0.76	6.42 <sup>b</sup> ±0.21
Rice	Control	70.84 <sup>a</sup> ±0.25	73.08 <sup>a</sup> ±0.75	73.72 <sup>a</sup> ±0.56	12.36 <sup>a</sup> ±0.35
	CV50°C	72.82 <sup>bc</sup> ±0.64	74.65 <sup>bc</sup> ±0.78	75.12 <sup>cd</sup> ±0.60	11.84 <sup>a</sup> ±0.18
	CV60°C	73.59 <sup>c</sup> ±0.65	75.53 <sup>cd</sup> ±0.42	76.25 <sup>d</sup> ±0.28	10.84 <sup>a</sup> ±0.26
	MV50°C	73.98 <sup>c</sup> ±0.58	75.99 <sup>c</sup> ±0.21	76.38 <sup>d</sup> ±0.71	9.34 <sup>a</sup> ±0.29
	MV60°C	74.22 <sup>c</sup> ±0.34	76.07 <sup>c</sup> ±0.64	77.15 <sup>d</sup> ±0.92	8.42 <sup>b</sup> ±0.22

Valued expressed as mean ± standard deviations of three independent experiments (n=2). Distinct letters represent statistical differences for the mean values at (P>0.05).

flour are measured by DSC and summarized in Table 3.

The results show all thermal properties of control corn flour were similar to conventional heated and microwave heated corn flour. Stevenson *et al.* (2005) had found that the thermal properties measured for native corn starch and microwave irradiated corn starch with 20-35% moisture content are similar. Results from Table 3 shown the range of gelatinization temperature of rice control is 70.84 to 73.72°C which in agreement with the previous research by Puncha-arnon and Uttapap (2013) which found that the range of gelatinization temperature of native rice flour is 69.4 to 77.3°C. Whereas, the onset gelatinization temperature (To) of corn control is 61.60°C which similar with the finding of Lewandowicz *et al.* (2000) shown the (To) of native corn starch is 61°C. However, there are significances differences on thermal properties between rice control and heat treated rice flour. Microwave heated rice flour at 60°C have significantly higher To, Tp, and Tc. Conventional heating and microwave heating exert similar effect on the rice flour, as there are insignificant differences shown by values of To, Tp, and Tc between conventional heated sample and microwave heated sample. According to Bilbao-Sainz *et al.* (2007) stated that mechanism of gelatinization was the same under both microwaves and conduction heating.

The results from the Table 3 has shown there is an increasing trend for To (both corn and rice flour) which is in agreement with previous research reported by Stevenson *et al.* (2005), who found that microwave radiation had shown an increment on the To of microwave radiated corn starch. Lewandowicz *et al.* (2000) has reported that microwave radiation

evidences to shift the gelatinization transition of cereal starches to higher temperature compared to untreated starches. This finding is similar with the pattern of results shown from Table 3 which also found that gelatinization range of microwave corn and rice flour had been shifted to the higher temperature. The increases of onset gelatinization temperature in starch might due to the molecular reorganization in the granule which leads to a more stabilize structure. Previous studies have been suggested that elevated in gelatinization temperature was due to the associations of amorphous amylose or configuration in granular structure with greater stability (Lewandowicz *et al.*, 2000; Stevenson *et al.*, 2005; Donovan *et al.*, 1983). Whereas, there are significant decreased of enthalpy of gelatinization (ΔH) for corn and rice flour, microwave irradiated corn and rice flour at 60°C has shown a significant lower value which is 6.42 ± 0.21 J/g and 8.42 ± 0.22 J/g respectively. The results show is in accordance with the previous study by Lewandowicz *et al.* (2000) and Stevenson *et al.* (2005), who mentioned that enthalpy change of gelatinization decreased substantially for microwave irradiated corn and wheat starches. Enthalpy of gelatinization (ΔH) is correlated to the thermal loss of double-helical order of starch rather than that of the crystalline arrangement of double helices (Cooke and Gidley, 1992; Lee *et al.*, 2012). Marked decrease of enthalpy of gelatinization for heat treated starch indicated there is loss of molecular order within the starch granules.

#### Scanning electron microscopy of control and treated starches

The SEM micrograph of control, conventional heated 60°C, and microwave heated 60°C of corn

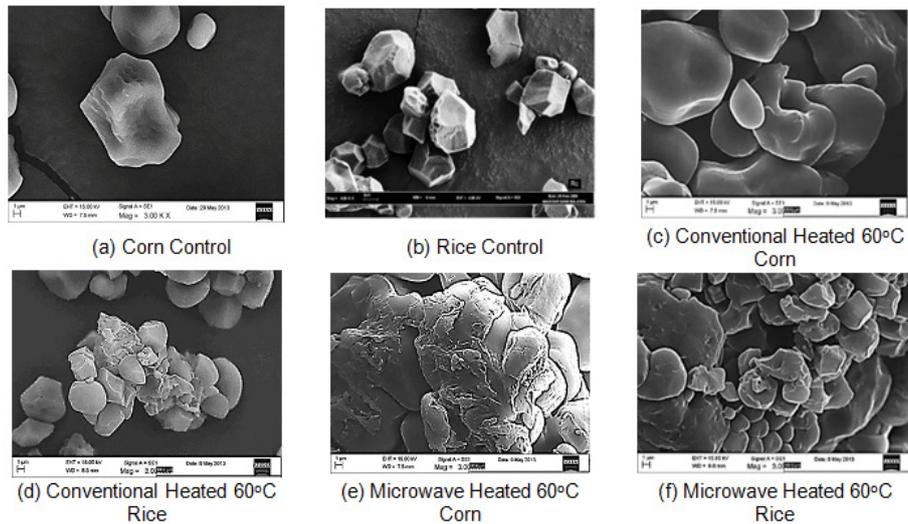


Figure 1. SEM micrographs (3000 $\times$ ) for (a) control corn, (b) conventional heated corn at 60 $^{\circ}$ C, (c) microwaved heated corn at 60 $^{\circ}$ C, (d) control rice, (e) conventional heated rice at 60 $^{\circ}$ C, (f) microwaved heated rice at 60 $^{\circ}$ C

and rice flour is shown in Figure 1. From the results, corn flour and rice flour had shown a significant variation in granular structures which varies in size and shape (Figure 1). The starch granules of corn show an angular shape whereas rice flour shows an angular and pentagonal shape. This observation is in accordance with the previous study by Singh *et al.* (2003) who investigated morphological of starches from different botanical sources. Size of the rice flour is much smaller than size of corn flour shown from the micrograph. According to Singh *et al.* (2003), average size of corn starch granules is varying from 1 to 20  $\mu$ m while rice starch granules are in the range from 3 to 5  $\mu$ m size. The extensive structural changes in conventional heated and microwave heated of corn and rice flour can be seen from the micrograph. Control of corn and rice flour has shown a completely undamaged starch structure as there are no heating treatment is applied on the starch. The pores, channels and cavities can be seen on starch granules of control flour under SEM. Jane (2009) has observed that under SEM, corn, wheat, barley, rye, sorghum and willet native starch granule appear to distribute with pores. For conventional heating treated corn and rice flour, damaged starch structure and eroded granules were observed in micrograph (Figure 1b and 1e). This indicated that conventional heating exert effect on the starch structure of corn and rice starch. However, the micrograph shows that there are only partially structural changes exist in starch granules of conventional heating treated but not every starch granules.

Little granule swelling may exist due to the conventional heating leads to the eroded granule. Microwave heated corn and rice flour show there are

cavity, pin holes, rougher surface observed under the SEM. Clumps can be seen from the starch granules on the microwave treated corn and rice starch. This observation indicated that there are certain granules had been ruptured, leaching out the amylose lead to the clumping of starch granules. Greater granule swelling may exist which might leads to the granules ruptured and clumps due to effect of microwave heating. As microwave energy capable of generate heat deep inside the penetrated medium of the starch granules by the molecular friction (Lewandowicz *et al.*, 2000).

#### X-ray diffraction of control and treated starches

X-ray diffraction diffractometry is using to reflect the characteristics of crystalline structure of starch granule. Atoms and molecules will interact with the electromagnetic waves of short wavelength result a diffraction pattern. Cereal starches reveal the typical A-type X-ray pattern, tuber starches reveal B-type and legumes show the mixed state of C-type (Singh *et al.*, 2003). X-ray diffraction patterns of control and heat treated of corn and rice flour are presented in Figure 2. Both corn and rice flour for control exhibited the typical A-type pattern with strong peaks at  $2\theta$  about 17.3 $^{\circ}$ , 17.7 $^{\circ}$  and 18.3 $^{\circ}$  for corn and 17.3 $^{\circ}$ , 18.2 $^{\circ}$  and 18.3 $^{\circ}$  for rice. According to Hwang *et al.* (2009), native corn starches showed a typical A-type crystal pattern with strong reflections at 15 $^{\circ}$  and 23 $^{\circ}$  of diffraction angle  $2\theta$ . Singh *et al.* (2007) has found that rice starches form non-waxy rice cultivars show typical A-pattern with strong reflection at  $2\theta = 15.1^{\circ}$ , 17.2 $^{\circ}$ , 18.1 $^{\circ}$ , and 23.2 $^{\circ}$ .

From the X-ray diffraction pattern shown by the heat treated corn and rice flour, microwave heated

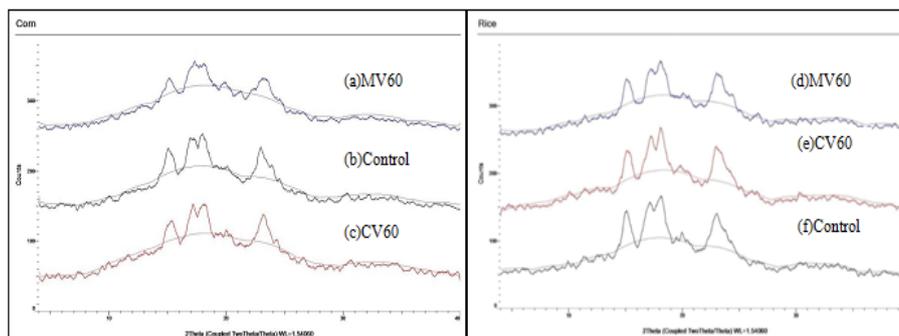


Figure 2. X-ray diffraction patterns of (a) microwaved heated 60°C corn, (b) control corn, (c) conventional heated 60°C corn (d) microwaved heated 60°C rice, (e) conventional heated 60°C rice, (f) control rice

corn flour show strong reflections at  $2\theta = 17.9^\circ$ ,  $18.3^\circ$ ,  $18.7^\circ$  than conventional heated corn flour with reflection at  $2\theta = 17.3^\circ$ ,  $18.3^\circ$ ,  $18.5^\circ$ , and control corn flour with reflection at  $2\theta = 17.3^\circ$ ,  $17.7^\circ$ ,  $18.3^\circ$ . This indicated that microwave heat treated corn flour appeared to slightly more crystalline character than conventional heat treated and control corn flour. Control rice flour and conventional heated rice flour show reflection at  $2\theta = 17.3^\circ$ ,  $18.1^\circ$ ,  $18.3^\circ$ , whereas microwave heat treated show reflection at  $2\theta = 17.3^\circ$ ,  $18.1^\circ$ ,  $18.5^\circ$ . This indicated that the crystalline character is relatively similar for control and heat treated rice flour. Heat treated corn and rice flour show the typical A-type pattern same as the control. Sharper x-ray diffraction peaks indicated the amorphous region has been disrupted by the heating. Higher peak intensities reflect greater crystallinity, while the lowest peak intensities reflect low granule crystallinity (Wani *et al.*, 2012). According to Tester *et al.* (2004), 0% of crystallinity representing fully amorphous material whereas 100% of crystallinity represent all the amorphous material has been eroded.

## Conclusion

Microwave heating treatment and conventional heating treatment exert the similar effect on the physicochemical and functional properties of corn flour and rice flour. The extent of the changes on physicochemical and functional properties is not similar between the microwave heating treatment and conventional heating treatment. The extent of the changes induced by microwave heating was greater than the conventional heating on the properties of starch. The microwave and conventional heating treatment was evidenced to cause several changes in functional, morphological and rheological properties of untreated starch. There are increasing of amylose content, particle size diameter, pasting temperature and gelatinization temperature as well as decreasing of pasting viscosity, swelling power and solubility of

all the heat-treated starches. Both heating treatment effect also lead to changes in the structure of starch granules, loss of birefringence, rupture of granules and leakage of constituent of starch. The different temperature effect which carried out at  $50^\circ\text{C}$  and  $60^\circ\text{C}$  for both heating treatment do not caused any significance difference effect on the functional and physicochemical properties on the starch different with the significant increment in particle size diameter. We hope that the output of this study will contribute more information about physical modification of starch, the microwave treatment on flour which is more environmental friendly to improve physicochemical and functional properties of native starch for greater utility in food uses in commercial scale.

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