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Pregelatinized Flours Using Extrusion Process: Effect of Barrel Temperature on Steady Shear, Dynamic Shear and Cohesiveness of Extruded Flours

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ABSTRACT

Effect of extruder barrel temperature and flour types on the rheological properties of extrudates produced from corn flour, rice flour and their blends with 30% potato starch were studied. The native flours were extruded under extrusion conditions at screw speed of 75 rpm, barrel temperature ranging from 80°C to 140°C and die size of 1.88 mm. Extrudates were dried at 50°C overnight and then the samples were ground to obtain extruded flours which further tested for steady shear, frequency sweep and cohesion test. The results showed pregelatinized flour have shear thinning behavior when processed under temperature ranging from 25°C to 95°C ($R^2 > 0.869$) and apparent viscosity equations obtained showed a decreasing E_a with increasing barrel temperature. All extruded flours tested have solid-like behavior on dynamic test and powder flow analysis showed that cohesiveness of the pregelatinized flour using extrusion process was not affected by the extruder barrel temperature.

Keywords: Extruded Flour; Steady Shear; Dynamic Shear; Apparent Viscosity; Cohesion Index.

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INTRODUCTION

Flour and starches have been used as food ingredient since ancient times in various food products due to their ability to thickened food products (Singh *et al.*, 2003). Although native flour and starches gave good thickening properties and textural properties in the product, they often cause problems such as having poor processing tolerance, low viscosity and poor stability. These problems lead to poor product quality as well as processing difficulties due to pressure drops in the processing pipeline (Arocas *et al.*, 2009, Singh *et al.*, 2007). To overcome this problem, researchers had come out with chemically modified starches that gave a better consistency and stability during processing pipelines as well as a more stable product (Jobling, 2004). However, the term “chemically modified” appears to be something unhealthy in consumers mind and food manufacturers are now searching for possible ways of having clean label by using physical modification methods which involve heating of flour or starch above gelatinization temperature with the presence of water (Neelam *et al.*, 2012). Extrusion process is not only includes the heating of the flour with the presence of water, but also involves the shearing of the flour by the screw while pushing the materials to the end of the extruder can be the one of the option.

In extrusion cooking there are several parameters that affect the degree of cooking or gelatinization of the flour, which led to different physicochemical properties of the pregelatinized flour. These include barrel temperature, screw speed, screw configuration, types of raw material and feed moisture content (Wu *et al.*, 2010, Tunick and Onwulata, 2006). In order to understand how the extruded flours behave in suspension under different processing temperature, the rheological properties of different flour extrudates were determined using steady and dynamic shear. Pérez *et al.* (2008) studied the steady shear rheological properties of maize-soybean mixture produced under at different extruder barrel temperature and moisture content, where the study found that higher extrusion temperature leads to lower viscosity of the dispersion. Chang and Cui (2011) studied the steady and dynamic shear of the extruded fenugreek gum solution at different concentration, the extrudate was found to have a shear thinning behavior and a weak gel was formed from the extrudate. These studies showed the importance of understanding effect of different extruder barrel temperatures on the rheological properties of the pregelatinized flour, and it showed the extruded flours showing shear thinning behavior and fits well with Arrhenius equation.

Other than looking only at the flow after the extruded flours was being formulated into the recipe, the powder flow properties of the extruded flours itself is also important for large scale industry where most of the powders are kept in the hopper. These information can be obtained by several tests using powder flow analyzer that mimics the mixing, handling and conveying of the powder throughout the processing line (Cuq *et al.*, 2011, Fitzpatrick and Ahrné, 2005, Teunou *et al.*, 1999). One of the important powder properties, cohesiveness, is the tendency of the powder particles to agglomerate together, where this might cause poor flow of powder (Janjatović *et al.*, 2012, Benkovic and Bauman, 2009). Many authors studied the effect of moisture, temperature and particle size on the powder flow properties of the food powder (Janjatović *et al.*, 2012, Benkovic and Bauman, 2009, Landillon *et al.*, 2008, Fitzpatrick *et al.*, 2007). Janjatović *et al.* (2012) assessed the powder flow properties of different soup concentrates which involve a mixture of food powders with different particle size while Landillon *et al.* (2008) reported the flow properties of raw wheat powders at different moisture content. However, there were little knowledge on the effect of extrusion and extrusion process parameter to the flowability properties of the powder.

The above-cited studies did not include native flours that are commonly used as food ingredient in the food industry, such as corn flour, rice flour and potato starch, as well as the effect of extrusion process to the flowability of the powder. Therefore, this study aimed to understand the steady shear, dynamic shear and the powder flow properties of these extruded flours produced under different extruder barrel temperature. Higher barrel temperature was expected to gelatinize better the flour, causing a lower apparent viscosity to the suspension, and have a better flowability. The information provided from this study gives an overview of the behavior of the extruded flour inside the hopper and the pipeline, which can help to minimize the problem of pressure drop during manufacturing food products as well as making a decision on the choice of extruded flour to be used as ingredient in their product formulation.

MATERIALS AND METHODS

Materials and sample preparation. The materials and samples were as prepared by Sue Shan *et al.* (2015). Corn flour (Gemini, Malaysia), rice flour (Gemini, Malaysia) and potato starch (Windmill, Holland) were purchased from the local market near Universiti Putra Malaysia, Selangor. Native flours: corn flour (CF), rice flour (RF), mixtures of corn flour to potato starch (CP; 70:30, w/w) and mixtures of rice flour to potato starch (RP; 70:30, w/w) were used as the raw material. Mixtures of 30% was commonly used by most of the researches to substitute the original formulation; hence it was chosen to be used in this study.

The samples were preconditioned to the moisture content of 25% w.b. The moisture contents of the samples were determined using a halogen moisture analyser (XM-120, Precisa Gravimetrics AG, Switzerland). Preconditioning of the samples was done using method described by Yu *et al.* (2012) with some modification based on preliminary testing. The flour samples were sprayed with water and homogeneously mixed by using a mixer (Alfa KB-502 Cake Mixer, Taiwan), then kept in the refrigerator at 4°C for 40h to ensure proper moisture distribution prior to extrusion process.

Starch, protein, fat and amylose/amylopectin content determination. The protein from the flour samples were removed by using the method Patindol *et al.* (2003) with slight modification. Flour sample of 1.5g was suspended in 6 mL of 0.05 N NaOH at 4°C for 1 h with shaking. The sample was then spun at 250 x g (Model CR412, Jouan Co., Saint-Herblain, France) for 5 min, then the supernatant and the top sticky protein layer poured off. The steps were repeated for 3 times and the sample was agitated overnight at 4°C in 6mL of 0.05N NaOH. The sample was suspended in deionized water, neutralized with HCl, and washed three more times with deionized water, rinsed with methanol, and lyophilized. The fat content of the flour was removed using Soxhlet method. The protein and fat content were done by using AOAC 981.10 and AOAC 991.36 (AOAC, 1999). Protein contents were determined by modified Kjeldahl method and the conversion factor of the nitrogen used in this study was 6.25 while the fat contents were determined by Soxhlet method.

Amylose contents of the flour samples were determined using method according to the method of Williams *et al.* (1970). Flour sample of 20mg were added with 10 mL of 0.5N KOH and dispersed using magnetic stirring bar. The sample was then diluted to 100 mL. About 10mL of the solution was added with 0.5 mL 0.1N HCl, followed by 0.5 mL iodine reagent and make up to 50 mL. After 5 minutes, the absorbance of blue color was measured at 625 nm using spectrophotometer (Genesys20, 4001/4, Thermo Scientific, USA).

Extrusion process

The extrusion process was carried out by using a Brabender single screw stand-alone extruder (KE-19/25 D, Brabender, Germany). The variables that were fixed in this study was the screw design, spiral screw with the compression ratio of 1:1, round die with the diameter of 1.88 mm and screw speed at 75 rpm. The flour samples were extruded under the same barrel temperature for all zones, and for each run the temperature was set at 80°C, 100°C, 120°C and 140°C. Each experiment set was conducted in triplicates. After extrusion process, the extrudates were dried in a cabinet dryer (SMA-112, Smoke Master, Japan) at 50°C for 16h. The moisture contents of the extrudates were determined using a halogen moisture analyzer (XM-120, Precisa Gravimetrics AG, Switzerland). The samples were packed in high density polyethylene (HDPE) plastic bag and kept at room temperature until further analysis.

Extruded flour preparation

The extrudates were grinded until they pass through a metal sieve with mesh size less than 425 µm using Super-g Mixer Grinder (KM501, Premier, India). The extrudate powder were then packed in high density polyethylene (HDPE) plastic bag and kept at room temperature until further analysis. The samples were coded using the initial of the flour type and followed by the barrel temperature used. For example, rice flour extruded at 80°C coded as RF80, corn flour extruded at 100°C coded as CF100, corn flour with potato starch extruded at 120°C coded as CP120 while rice flour with potato starch extruded at 140°C coded as RP140.

Preparation of extruded flour dispersion

For steady shear analysis, the extruded flour suspensions were prepared by dispersing 0.6g of the extruded flour in 10 mL of water at room temperature (6 % w/w). The samples were mixed for 30 min at 125 rpm using magnetic stirrer hotplate heating surface (Fisherbrand, Fisher Scientific, USA). Then, the solution was transferred into a water bath at 95°C for 30 min and measured for the steady shear rheological properties.

For stress sweep and frequency sweep analysis, the extrudate gels were prepared in a plastic testing tube with cap by dispersing 1.00 g of the extruded flour in 4.00 g of water at room temperature (25 % w/w). The samples were mixed for 1 min under high speed vortex mixer (FineVortex 4S, FinePCR, Korea) and held for 1 hour prior to analysis to ensure proper hydration of the extruded flour. The samples were capped to prevent moisture loss during holding time.

Particle size distribution determination

Particle size distribution of the extruded flour was determined using Malvern Mastersizer 2000 equipped with Scirocco 2000 dry powder dispersion unit (Malvern Instruments Ltd, Worcestershire, UK). The particle size range measured was from 0.020 to 2000 µm. The measurement were done under the condition as below; refractive index of 1.5, 1 measurement cycles of 10 s each, feed vibration rate at 12 % and the air flow pressure at 0.75 bar. All samples were measured 5 times and the average particle size distribution. The particle size of extruded flour was characterized using d(0.1) which indicates 10% of the volume distribution is below the value, d(0.9) which indicates 90% of the volume distribution is below the value, volume median diameter d(0.5) which indicates 50% of the distribution is above and 50% of the distribution is below that value, and the volume weighted mean d(4,3).

Steady shear test

Steady shear test were done according to the method mentioned by Staroszczyk *et al.* (2013) Manoi and Rizvi (2009) with some modifications. The sample was loaded into the rheometer (Haake Rheostress 6000, Thermo Scientific, USA) using parallel plate geometry PP35Ti with 1 mm gap. Extruded flour suspensions were scanned at 25°C, 35°C, 50°C, 75°C, 85°C and 95°C at the range of shear rate of 10-200 s^{-1} (the range for mouth chewing and general processing condition). All rheological methods were done in duplicates and the shear stress and shear rate plotted by the system were recorded. Power law model, Eq. (1) was used to describe the steady shear rheological properties of the samples,

$$\sigma = K\dot{\gamma}^{n} \qquad \text{or} \qquad \eta = K\dot{\gamma}^{n-1} \tag{1}$$

where σ is the shear stress in Pa, $\dot{\gamma}$ is the shear rate in s^{-1}, K is the consistency coefficient in Pa.s^n and n is the flow behavior index. By using the K and n obtained, apparent viscosity at 100 s^{-1} (η_{100}) was calculated.

The effect of processing temperature (25°C, 35°C, 50°C, 75°C, 85°C and 95°C) on the consistency coefficient of the extruded flour were determined using Arrhenius equation, Eq. (2),

$$\mathrm{K} = \mathrm{A}e^{\mathrm{E_a}/\mathrm{RT}} \tag{2}$$

$$\ln \mathrm{K} = \ln \mathrm{A} + \frac{E_a}{\mathrm{RT}} \tag{3}$$

where K is the consistency coefficient in Pa.s^n, A is the Arrhenius constant in Pa.s^n, T is the absolute temperature in Kelvin, R is the gas constant (8.3144 J.mol^{-1}.K^{-1}) and E_a is the activation energy (kJ.mol^{-1}) and was obtained by using the value of the slope plotted according to Eq. (3). Higher activation energy showed higher dependency of the viscosity of the suspension on the processing temperature.

Frequency sweep test

Frequency sweep test was run according to Wu *et al.* (2010) with some modification. The sample was loaded into the rheometer (Haake Rheostress 6000, Thermo Scientific, USA) with parallel plate geometry

PP35Ti. Samples were scanned at 25°C under frequency of 1-100 Hz and shear stress of 0.1 Pa which in the linear viscoelastic range (LVR) for all the extrudates according to the stress sweep test.

Cohesiveness test

Cohesiveness test was done according to Benkovic and Bauman (2009) and Janjatović *et al.* (2012) with slight modifications. The cohesiveness of the extruded flour was determined using TA.XT Plus with Powder Flow Analyzer (PFA) attachment (Stable Micro Systems, Surrey, UK). The PFA attachment used was small vessel attachment, with a cylindrical glass vessel (80 mm height and 25.5 mm diameter) with a steel rotor blade (23 mm diameter and 10 mm height) which able to move upwards, downwards and rotate clockwise and anti-clockwise (Landillon *et al.*, 2008). The data were processed using Texture Exponent 32 software (Stable Micro Systems, Surrey, UK) provided by the equipment supplier. Extrudate powders were filled into the vessel until 70 mm height (or 35 mL).

Cohesion test was run following the procedure suggested by the powder flow analysis software. The test was carried out by two initial conditioning cycles to ensure proper distribution of the powder. Then, the level of the powder was checked and the weight of the sample was recorded. The test continued with a downwards move using a “cutting˝ action until the base followed by an upward movement where the powder was lifted and the force of the powder on the vessel base was measured. After the cycle, the weight of sample was required to be entered into the software and calculation of the cohesion index was done by dividing the negative area of the graph obtained (which is the area of the force for the powder on the vessel base, also known as the cohesion coefficient) with the weight of the sample. The bulk density of the samples was calculated using Eq. (4).

$$\text{Bulk density, kg. m}^{-3} = \frac{\text{weight of samples (kg)}}{\text{volume of sample (m}^3\text{)}} \tag{4}$$

Statistical analysis

Data obtained for steady shear test were processed using “Solver” tool in Microsoft Excel (Microsoft Excel 14.0.7116.5000, Microsoft Corporation, USA). Average values of the plot of frequency sweep test were calculated using Microsoft Excel (Microsoft Excel 14.0.7116.5000, Microsoft Corporation, USA) and plotted using scatter plot mode.

RESULTS AND DISCUSSION

Proximate analysis

The starch, protein, fat, amylose and amylopectin content of the raw materials were tested and the results were shown in Table 1. Starch contents of the raw materials were significantly different. The starch, amylopectin and protein contents of raw materials are significantly different among them. CP contains the highest starch content while RF has the lowest starch content ($p \le 0.05$), and RF contains the highest protein content while CP has the lowest protein content ($p \le 0.05$). The fat and amylose contents of the raw materials are significantly ($p \le 0.05$) different among them except between CP and CF ($p > 0.05$). CF has the highest amylose content (16.0 % ± 0.0012), followed by CP (15.9 % ± 0.30), RF (12.7 % ± 0.30) and RP (11.2 % ± 0.14). For amylopectin content, RP has the highest amylopectin (74.7 % ± 0.14) followed by CP (71.1 % ± 0.28), CF (70.0 % ± 0.0054) and lastly RF (60.3 % ± 0.36). All these information is mainly due to the botanical origin of the raw material and can be one of the contributors to the properties of the extrudate.

Steady shear

All of the extrudate suspensions were well fitted into power law model, Eq. (1), with the R^2 ranging from 0.869 to 0.999. It showed shear thinning behavior with the n value less than 1 (ranging from 0.37-0.90), and with the K value ranging from 0.01 to 3.59 Pa.s^n.

To obtain the activation energy of the extrudate suspension, which showed the temperature dependency of the suspension, Arrhenius equation (Eq. 2 and 3) was used by fitting the K value into the equation under different processing temperature and results are shown in Table 2. The extrudate powder showed dependency on rheometer processing temperature, where increased in the rheometer processing temperature reduced the viscosity of the extrudate samples.

Effect of processing temperature

The effect of processing temperature on flow behavior of the extrudate suspension can be observed through the viscosity of the extrudate suspension at the shear rate of 100 s^{-1} as presented and range of temperature in Table 2. In this study, the processing temperature covers from room temperature (25°C) to high temperature (95°C). It is important to study the effects of temperature on the flow behavior of extrudate suspensions as they are processed and stored under various temperatures (Kim and Yoo, 2009).

The results showed that increased in processing temperature reduced the apparent viscosity of the extrudate, which agrees with many researchers. Chun and Yoo (2004) reported that sweet potato flour dispersion has decreasing apparent viscosity with increasing processing temperature from 25°C to 70°C. Moreira *et al.* (2012) reported that chestnut starch dispersion decreased at higher processing temperature, from 25°C to 70°C. This phenomenon can be related back to thermal expansion which cause increased in the intermolecular distance of the starches, making the flow easier and smoother (Constenla *et al.*, 1989).

Activation energy (E_a) in Arrhenius equation for rheological tests indicates the sensitivity of the extrudate suspension towards temperature. E_a of the extrudate powder ranged from 4.20 – 24.96 kJ.mol^{-1}. However, not every sample showed good fit in Arrhenius equation, such as RP140, CF100, CF120 and CF140, and they were not shown in Table 2. Holdsworth (1971) has reported that E_a can be related to the temperature sensitivity of the samples' viscosity, where higher E_a showed higher temperature sensitivity of samples' viscosity. The inconsistent trend of CF and RP for the activation energy caused the poor determination coefficient of the flour (R^2 = 0.381 – 0.528). This poor regression showed that the viscosity of CF and RP extrudate is not strongly affected by processing temperature. Similar result was shown by Kim and Yoo (2006) on 0.8% xanthan gum dispersion where they suggested that the dispersion was not greatly influenced by processing temperature. CF and RP have the higher starch content based on the proximate analysis done by Sue Shan *et al.* (2015). The study of Bortnowska *et al.* (2013) reported that increase in starch content decreases the E_a and decreases the temperature dependency of starch suspension. Their finding explained the poor fit in Arrhenius equation of RP140, CF100, CF120 and CF140 in this study. Hence, for those samples which did not have a good fit for Arrhenius equation, it is still a risk for using them in the industry as the viscosity of the suspension during the changes in processing temperature is unpredictable and it may still cause pressure difference and flowability problem in the processing pipelines.

Effect of barrel temperature and flour type

There were not much study done on the effect of the barrel temperature on the rheological properties of extrudate suspension. When looking at the apparent viscosity (η_{100}) showed in Table 2, the viscosity decreased with increasing barrel temperature. Higher barrel temperature provides more energy and higher starch gelatinization (Yeh *et al.*, 1999) which led to lower viscosity of the extrudate suspensions (Wu *et al.*, 2002).

The rheogram on effect of barrel temperature on different flour type as shown in Fig. 1, showed that increased in barrel temperature from 80°C to 120°C reduced the shear stress of the RF and RP extrudate suspensions. However, there was only little difference for CF and CP extrudate suspensions, largely due to the starch composition of CF and CP are insignificant ($p>0.05$). Looking into the viscosity of the extrudate suspension, the viscosity reduced with increasing barrel temperature for all extrudates. This indicates flour type dominants the effect of extrudate suspensions' rheological properties. The gelatinization temperature of CF, RF and potato starch were 69.4°C, 76.2°C and 62.6°C respectively (Singh, 2010). RF and RP with higher gelatinization temperature showed larger differences between the viscosities of the samples. The reason behind this is that RF and RP have a shorter residence time as reported in Sue Shan *et al.* (2015), and the barrel temperature is 80°C limits the gelatinization. When barrel temperature increases, the flour gets more heat to gelatinize and produced a better gelatinized extrudate. When the extrudates were made into suspensions, the shear stress of better gelatinized extrudate suspensions (higher barrel temperature) became lower.

When looking into the activation energy, extrudate powder of rice flour (RF) and corn flour with potato starch (CP) showed decreased in activation energy with increasing barrel temperature. This indicates that the extrudate suspension is still temperature dependent, but the effect of rheometer processing temperature is less when the extrudate is processed under higher barrel temperature. The results shown for RF and CP in this study is in line with the study of Staroszczyk *et al.* (2013), where they reported that native potato starch showed higher E_a compared to irradiated potato starch, and longer irradiation showed lower E_a. As for rice flour with potato starch (RP) and corn flour (CF), there were no special trends on the activation energy when compare to barrel temperature. This is due to the poorly fitted Arrhenius equation of the samples.

The effect of flour type on the rheological properties was shown in Fig. 1. Addition of potato starch in CF did not have much effect on the rheological properties of the extrudate samples. This is due to no significant difference of the amylose and content between CF and CP. Sample RF and RP showed different trends on the effect of flour type. At barrel temperature of 80°C, RF had higher viscosity and shear stress. As barrel temperature increased, the viscosity and shear stress of RP overtook RF. The gelatinization temperature of RF was found to be 66.00-78.04 °C (Singh *et al.*, 2003b), where barrel temperature of 80°C and having the shortest residence time as reported in Sue Shan *et al.* (2015) limits the gelatinization of the starch of the RF extrudates causing higher viscosity in RF80 than RP80. When barrel temperature increased, the gelatinization of RF increased and hence RP that contains higher amylopectin content showed greater apparent viscosity and shear stress when compared with RF.

Addition of potato starch to RF reduced the E_a, but this does not happened in CP. The reason behind this difference is due to CF and CP have no significant differences in both amylose and amylopectin content, whereas RP has significant higher starch content than RF. This was supported by the study of Bortnowska *et al.* (2013) where E_a of the sauces added with potato starch decreased with increasing concentration of starches. However, additional of potato starch in CP makes CP extrudate heat dependable which can be easily calculated and predictable for application, while the opposite way happened for RF/RP. Hence, CP and RF are more suitable to be use for industrial application.

Dynamic shear

The dynamic shear data was plotted by log (G´, G˝) versus log ω, and the magnitude of the slopes (n´, n˝) intercepts (K´, K˝) and R^2 are calculated based on the equation below, Eq. (5) and (6):

$$G' = K' (\omega)^{n'} \tag{5}$$
$$G'' = K'' (\omega)^{n''} \tag{6}$$

The n´ and n˝ indicate the frequency dependency of the extrudate sample (Wu *et al.*, 2010). The results in Table 4 shown that all extrudate gels showed decreased n´ and n˝ with increasing barrel temperature, which indicates the increase in frequency dependency of the extrudate gels.

The plot of log (G´, G˝) versus log ω of the extrudate powder gels at 25% (w/w) concentration (Fig. 2) showed that G´ and G˝ increased with increasing frequency, where this indicates the gels are frequency dependent. All of the gels showed higher G´ when compare with G˝ (except RF at higher frequency). This showed that all the gels exhibit a solid-like behavior. As for RF at the frequency of 628.3 rad.s^{-1}, the high frequency disrupts the gel stability and the extrudate powder becomes a viscous product instead of elastic product. Similar findings on the frequency dependency of the G´ and G˝ of extrudate samples were reported for various extrudate products, such as the study on extruded corn starch (Ditudompo *et al.*, 2013) and extruded flaxseed-maize blend (Wu *et al.*, 2010). The G´ of all extrudate powder gels were always higher than the G˝, where this indicates the pastes behaved more solid like a gel. In addition, the study of Wongsagonsup *et al.* (2014) on cross-linked tapioca starch and Juszczak *et al.* (2013) on modified starches had similar trend of storage and loss modulus, which indicates the extruded flour has similar properties with modified starches.

The trend of G´ and G˝ for RF and CF were similar, where the overall G´ at 80°C is the lowest, while it did not vary much for 120°C and 140°C. This finding agrees with the finding of Wu *et al.* (2010). Their study explained that temperature plays an important role in forming the network of product thermally. At lower temperature, the extrudate formed flexible network, but at higher temperature which 140°C, the internal hydrogen bonds of the sample were disrupted and caused swelling of starch granules, which led to a lower strength gel. As for RP and CP, the trends are similar, where at lower extrusion temperature (80°C-100°C), frequency has less effect on the gel and the gel has higher storage modulus, whereas at higher extrusion temperature (120°C-140°C), the G′ was highly affected by the frequency with a lower storage modulus. This condition can be related back with the study of Sue Shan *et al.* (2015) on the WSI of the samples. For RP80 and RP100, the WSI of the extrudate samples are significantly lower than RP120 and RP140. The very low WSI makes the extrudate gel maintain more as a solid (elastic) gel rather than a liquid (viscous) gel. As for CP, similar observation like RP can be found, with a less diverse effect. Effect of flour type on the G′ and G″ can be observed in Fig. 2 as well. Addition of potato starch found to lower the graph by one log, and increased the low temperature extrudate gel G′ and G″. Potato starch itself possessed a lower G′ as reported by Li and Yeh (2001) and thus the overall storage modulus decreases.

Particle size distribution and bulk density

The particle size distribution and the bulk density of different extrudate powders have median particle size, d(0.5) of the extrudate powder ranged from 14.26 µm to 60.98 µm, while the mean particle size, d(4,3) of the extrudate powder ranged from 40.81 µm to 92.88 µm. In general the particle size distribution for these extrudate powders were widely spread, which indicates that they were not homogeneous in size (Teunou *et al.*, 1999). Flour type and different barrel temperature showed no significant effect on the bulk density of the extrudate powders ($p > 0.05$). Therefore, the effect of bulk density on the flowability properties of the extrudate samples could not be determined. This is similar with the study of Benkovic and Bauman, (2009), whereby the bulk density of the infant formula samples have no significant difference and hence bulk density were not the factor for the different flowability properties.

Extrudate powders are potential ingredients for baby food, and based on Codex Alimentarius (2011), there are no specific particle size requirement for baby food. Bhandari *et al.* (2013) suggested that different particle size distribution are required for each individual product, where fine particle flour might cause problem during transportation and coarse particle flour might cause unpleasant eating quality of the end product. Dried-milled corn flour was found to be in the particle size range of less than 170 µm, and all of the corn based extrudate powder in this study fell below this range (Kent and Evers, 1994). It is suggested that pregelatinized flour or starch with 90% of the particle size less than 150 mesh (equivalent to 105 µm) are easily well dispersed (Irving *et al.*, 1971). Based on the results, only RF100, CP80 and RP80 met the suggestion above.

Cohesiveness

The cohesion coefficient at 50mm.s^{-1} and cohesion index of the extrudate powders were shown in Table 4. The cohesion coefficient of the samples at 50 mm.s^{-1} had significant differences ($p \leq 0.05$) between different samples with same volume used. These values contribute to the cohesion index when they were divided with the weight of the sample (Landillon *et al.*, 2008). The cohesion indexes for all extrudate powders were below 11, which indicate that all of the extrudate powders are free flowing product (Ramavath *et al.*, 2013, Benkovic and Bauman, 2009, Landillon *et al.*, 2008).

Barrel temperature showed no significant effect ($p > 0.05$) on the cohesion coefficient at 50 mm.s^{-1} for extrudate powder of corn flour, rice flour and blends of rice flour with potato starch. As for CP extrudate powder, the lowest cohesion coefficient at 50 mm.s^{-1} was found at the barrel temperature of 80°C with the mean particle size of 29.08, while the highest cohesion coefficient is at 100°C with the mean particle size of 42.83. This result is similar with the finding of Benkovic and Bauman (2009) in infant formula (milk based) samples where higher mean particle size led to higher compaction coefficient, but opposed with the result of Landillon *et al.* (2008) where smaller particle size led to higher compaction coefficient in wheat powder. The cohesion coefficient of powder not only depends on the particle size, but also the moisture content, relative humidity and environmental temperature during the test. The tests are conducted under the same environment; hence the difference on cohesion coefficient is due to the processing condition of the extrudate. Different flour types also showed significant differences in the cohesion coefficient ($p \leq 0.05$), but only at barrel temperature of 80°C. Sample RF80 showed the highest cohesion coefficient, followed by RP80, CF80 and CP80. The results showed that an addition of potato starch in the sample reduced the cohesion coefficient value. This is due to the high amylopectin content in potato starch which makes the extrudate powder more adhesive rather than cohesive (De Jong *et al.*, 2011), and the result showed that RF has the least amylopectin.

Rice grain was known to be highly cohesive, and hence its flour showed highest cohesion coefficient as in this study (Marianski and Marianski, 2011).

As for the cohesion index, which is the cohesion coefficient divided with the weight of the sample used, barrel temperature showed no effect on the cohesion index for all types of flour, but different flour showed significant different on the cohesion index ($p \leq 0.05$). Rice flour extrudate powder showed significantly higher cohesion coefficient ($p \leq 0.05$) when compared to corn flour extrudate powder and corn flour with potato starch mixture. This suggested the difference of cohesion index for the extrudate powder in this study that rice flour extrudate has significant higher cohesion index than corn flour extrudate.

CONCLUSIONS

All of the extrudate powder suspensions showed shear thinning behavior when processed under temperature ranging from 25°C to 95°C with R^2 more than 0.869 when fitted into power law model. CP and RF which are well fitted to Arrhenius equation and have decreasing E_a with increasing barrel temperature will be a good thickener for industrial application as they can predict the apparent viscosity of the product in the pipeline to avoid possible pressure drop and viscosity changes in the pipeline. As for gelling, RP and CP at 80°C and 100°C have less frequency dependency where they can maintain the gel even at high frequency, with a moderate storage modulus. The cohesiveness of the extruded flours was not affected by the extruder barrel temperature.

Table 1. Starch, Protein And Fat Content Of Samples

Raw Material	Starch (%)	Protein (%)	Fat (%)	Amylose (%)	Amylopectin (%)
CF	86.03^b±0.0066	0.29^c±0.00	0.28^c±0.0097	16.0^a±0.0012	70.0^c±0.0054
RF	72.99^d±0.057	9.24^a±0.012	1.39^a±0.040	12.7^b±0.30	60.3^d±0.36
CP	86.93^a±0.058	0.16^c±0.00027	0.17^c±0.020	15.9^a±0.34	71.1^b±0.28
RP	85.60^c±0.00	6.72^b±0.044	1.13^b±0.019	11.2^c±0.14	74.4^a±0.14

*Data shown is mean ± standard deviation

*Different letters (a - d) after value indicates significant differences (p≤0.05) down the column.

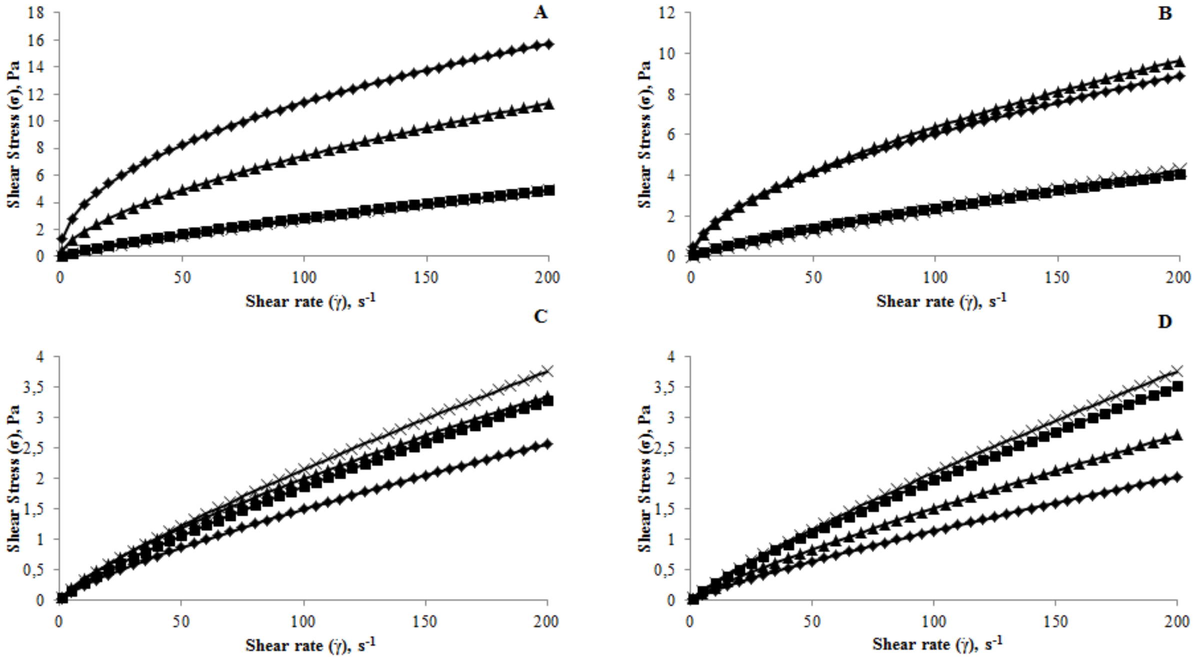

Figure 1 Rheograms of extruded flours: RF (♦), RP (▲), CF (■), CP (×) at barrel temperature, (A) 80°C, (B) 100°C, (C) 120°C, and (D) 140°C

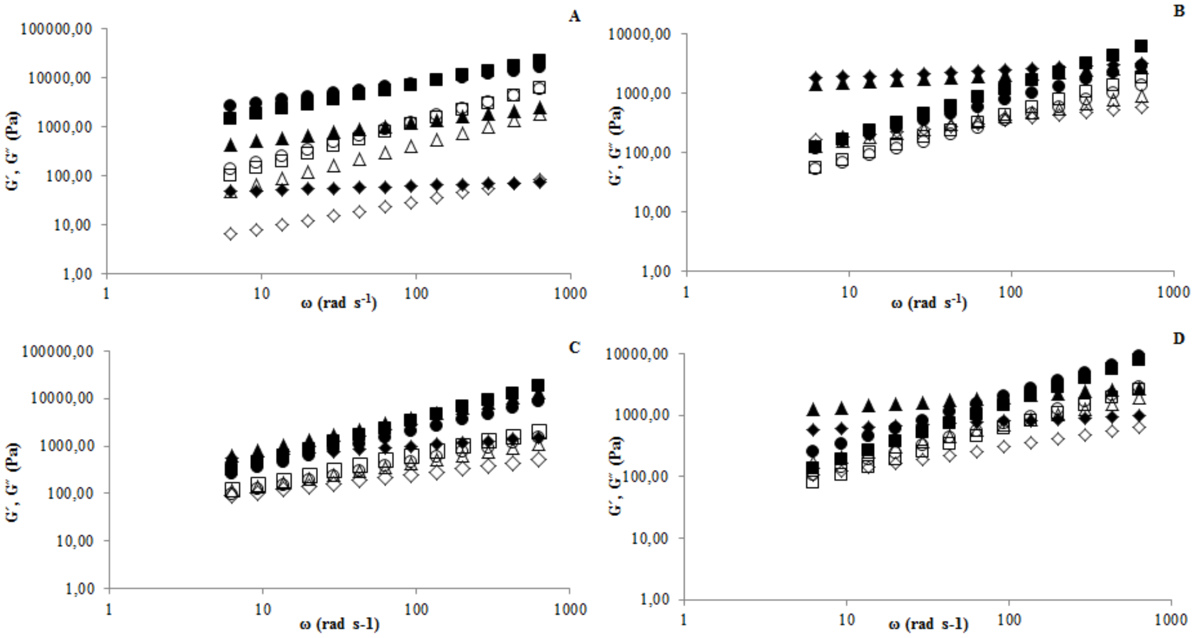

Figure 2. Storage Modulus (G´) And Loss Modulus (G˝) At 25°C Of Extrudate Powder Produced At Barrel Temperature Of 80°C (♦, ◊), 100°C (▲, Δ), 120°C (●, ○), 140°C (■, □) FOR (A) RF, (B) RP, (C) CF, AND (D) CP.

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