Rheological Properties of Cassava Starch-Kaffir Lime Essential Oil Film Solution

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Abstract—**Food packaging from synthetic polymers are not easily degraded which later can cause environmental issues. On the other hand, food products still need protection as these products are easily deteriorated by oxidation and microbial growth. As an alternative to the conventional food packaging, antimicrobial edible film will extend the shelf-life of food and minimize the use of synthetic plastic. The incorporation of kaffir lime essential oil as antimicrobial agent in edible film formulation has not been studied yet. The rheology of the formulation and the thermal properties of the produced film are important in determining the final characteristics of the film. The rheological properties were studied at temperature range from 45°C to 85°C using Anton Paar MCR 300. The rheological properties specifically the viscosity of the cassava film formulation is found to be decreases as the formulation is heated from temperature ranging 45°C to 85°C but does not proportional with the concentration of kaffir lime essential oil in the cassava film solution. The minimum viscosity of 0.23 Pa.s obtained is at temperature 85°C and concentration of kaffir lime essential oil at 0.6% v/v, while the highest viscosity 1.31 Pa.s obtained was at 0.0% v/v of kaffir lime essential oil. The pattern of the shear stress to shear rate graph conclude that this solution was a shear thinning model. Also, the flow behaviour of the film solution have a pseudoplastic behaviour and the activation energy evaluated is increases as the solution viscosity increases.**

Keywords— Cassava, Edible film, Food packaging, Kaffir lime essential oil, Rheological properties.

I. INTRODUCTION

Food packaging from synthetic polymers are not easily degraded which can cause environmental pollution. On the other hand, food products still need protection as these products are easily deteriorated by oxidation and microbial growth. As an alternative to the conventional food packaging, antimicrobial edible film will extend the shelf-life of food and minimize the use of synthetic plastic. The incorporation of kaffir lime essential oil as antimicrobial agent in edible film formulation has not been studied yet. The rheology of the formulation is important in determining the final characteristics of the film. Thus, this research will investigate these properties at different film formulation.

The extent of the study is to formulate an edible cassava film incorporated with kaffir lime essential oil for food packaging. Kaffir lime essential oil is extracted from kaffir lime peel by using hydro-distillation method. The formulation of starch film is varied in terms of kaffir lime essential oil concentration and the amount of glycerol and starch powder. For the rheology behaviour, apparent viscosity of varied cassava emulsions is determined at 45°C to 85°C by using rheometer.

In this study, the edible film formulation consists of cassava starch as polysaccharides, glycerol as plasticizer, water and kaffir lime essential oil as antimicrobial agent. The application of edible film is a promising way to the food technology industry as it preserves the whole fruits (Han *et al.,* 2006). The films act as barriers to water loss and gas exchange, controlling the transfer of moisture, oxygen, carbon dioxide, lipids and flavour components, with similar effect to that promoted by storage under controlled or modified atmosphere (Vargas *et al*., 2008). For this study, the effect of temperature on the formulation of edible film will be determined based on rheological properties. The information obtained can be used for designing and process evaluation, process control, and consumer acceptability of the product.

Rheological properties can be determined by the relationship of the stress applied on a material and the subsequent deformation as a function of time. The main parameters that involve in this study is stress and strain. Stress can be defined as a force applied per unit area and the type of stress is determine by the direction of force on the surface which normally can be either an extension or compression force while, strain is the extent of the deformation. Viscosity is a measure of resistance to flow of the fluid and defined as the ratio of applied shearing rate to the rate of shear strain (shear rate).

Interrelated to the shear viscosity, the elongational viscosity can also be defined by η (= σ / ε). The above definitions are difficult but they have only a limited applicability. This is because the ratio of stress to strain for solids and liquids is independent of strain and constant (independent of strain rate) only as γ (γ.) → 0. Generally, there are many forms of stress-strain curves and their shape can be identified with brittleness, toughness, etc (Malkin & Isayev, 2017).

Pseudoplasticity is the decreasing slope of the shear stress and strain rate relationships for polymer solution which is part of the fluid characterisation. Low molecular weight and swollen particulate systems sometimes give Newtonian behaviour (η independent of γ.) or dilatancy ("shear thickening"), respectively. It is convenient to determine the value of η over a extensive range of γ. for this kind of solutions and can be measure at both steady and oscillatory shear which are sometimes referred to as 'rheogoniometers' or 'mechanical spectrometers' (Picout *et al*., 2011).

The study of rheological behaviour of edible film is important for process scale-up to ensure that processing requirements and machinability issues can be accurately designed. Rheological properties of composite starch emulsions show pseudoplastic behaviour when (η < 1), while the Power law rheological model provided a satisfactory behaviour fit enough to experimental data (γ^2 > 0.96). Plasticizer and lipid addition to corn and amylomaize starch suspensions decreased the flow behaviour index and increased the consistency index (Embuscado & Huber, 2009). Apparent viscosity of corn starch suspensions decreased with both plasticizer and lipid addition. Measurement of viscosity is important. Spraying requires low viscosity, while immersion requires a higher viscosity coating solution. In formulation of composite biopolymer films, it is important to characterize the miscibility of biopolymers and interactions that may occur between them, since these attributes ultimately influence film microstructure.

II. METHODOLOGY

A. Materials

Fresh kaffir lime was purchased from Makassar Fresh Market in SS15 Subang Jaya. Cassava starch was purchased from nearby supermarket. Glycerol was obtained from the chemistry laboratory of Faculty of Chemical Engineering, UiTM Shah Alam.

B. Preparation of kaffir lime essential oil

The flavedo of kaffir lime was separated by peeling. The fresh peel flavedo then submitted to hydro distillation using a Clevenger-type apparatus. The peel is extracted with 5 L of water until there are no more essential oil obtained. The essential oil is then collected in a brown color vial and dried over 2g of anhydrous sodium sulphate and stored at 4°C.

C. Preparation of cassava film formulation

The preparation of cassava film formulation incorporated kaffir lime essential oil was adopted from Utami *et al.* (2018). 5 g of cassava starch was mixed with 100 mL of distilled water and heated at 60°C until gelatinized. 2 mL of glycerol was then added into the gelatinized formulation and heated at 60°C for 30 minutes. After 30 minutes, the formulation was added with 0.2 mL of kaffir lime essential oil and let cooled. The procedure was repeated by adding different volume of kaffir lime essential oil at 0.6 mL, 1.0 mL and 1.4 mL. Also, cassava film formulation without addition of essential is prepared as a control formulation. Every formulation is label as 0.0% v/v, 0.2% v/v, 0.6% v/v, 1.0% v/v and 1.4% v/v based on kaffir lime essential oil concentration.

D. Determination of cassava film solution viscosity

The viscosity of cassava film formulation is determined by using modular compact rheometer (Anton Paar MCR 300). The range of temperature used were between 45°C to 85°C with 31 measurement points at the rate of 2°C/min. The viscosity data of all 5 sample were all calculated and compiled into a graph of viscosity vs temperature.

RESULTS AND DISCUSSION

A. The effects of temperature on the cassava film formulation viscosity

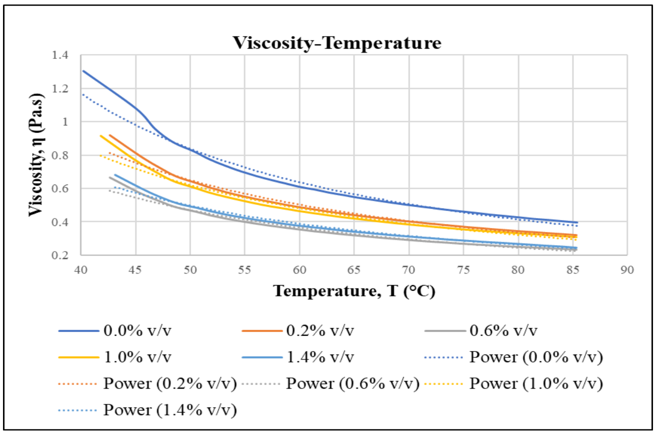

Fig. 1: Effect of temperature on cassava film formulation viscosity

The range of temperature used in this research was in between 45°C to 85°C. As the formulation heated from 45°C to 85°C the viscosity of the cassava film formulation is decreases from 1.31 Pa.s to 0.23 Pa.s. As seen in Figure 4.6, the viscosity of all film solution was decreases as the solution heated over time. The lowest viscosity of 0.23 Pa.s data obtained is at temperature 85.30°C whereby the concentration of cassava solution film was 0.6% v/v. On the other hand, the highest viscosity read was at 1.31 Pa.s, when there was no contain of kaffir lime essential oil in the solution.

The concentration of kaffir lime essential oil in the solution did not influence to the decreases of viscosity of the solution as the concentration increases. Based on Figure 4.6, it can be observed that the viscosity of cassava film solution was slightly the same for the concentration of 0.2% v/v and 1.0% v/v which the viscosity value obtained was in between 0.32 Pa.s to 0.77 Pa.s. Meanwhile, for cassava film solution at concentration of 0.6% v/v and 1.4% v/v was only had change insignificantly where the range of viscosity obtain was in range of 0.23 Pa.s to 0.59 Pa.s. The order of concentration of kaffir lime essential oil from the lowest peak viscosity to the highest peak viscosity was; 0.6% v/v, 1.4% v/v,1.0% v/v, 0.2% v/v and 0.0% v/v.

B. Relationship of shear rate and shear stress

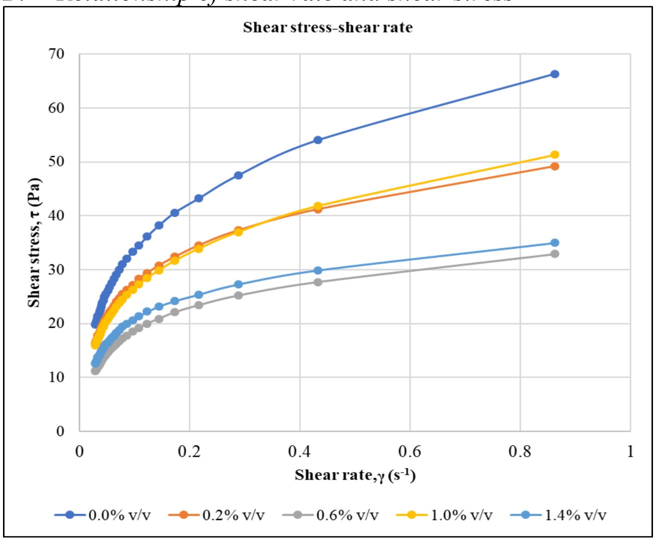

Fig. 2: Shear stress as a function of shear rate for different solution concentration

As in Figure 2, the relationship of shear stress and shear rate can be observed. The pattern of the shear stress to shear rate graph plotted conclude that this solution was a shear thinning model as the shear stress increases as the shear rate become longer.

C. Power law model of film solution

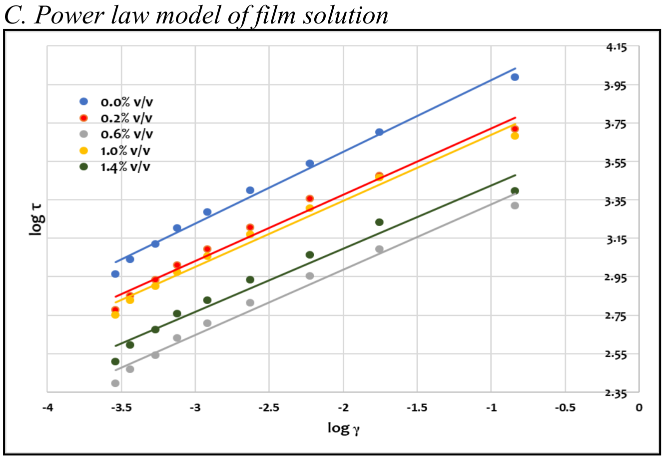

Fig. 3: Power law model

Table 1: K and n value from power law model

	0.0% v/v	0.2% v/v	0.6% v/v	1.0% v/v	1.4% v/v
K	77.1	58.5	39.1	56.4	39.2
n	0.37	0.34	0.34	0.34	0.34

Power law model as in Figure 3 was obtained from by log both shear rate and shear stress data. Then, the data will be linearized. K and n was obtained based on power law model equation.From Figure 3, K and n value of the film solution was obtained. K is the consistency coefficient while n is the flow behavior of the film solution. As in Table 1, the n obtained for all five different concentration of kaffir lime essential oil of cassava starch film solution is less than 1. From the literature, if the value of n is less than 1, the characteristic of film solution is pseudoplastic. All of the n value for cassava starch with the any concentration of kaffir lime essential oil is at 0.34. Meanwhile, for the cassava starch film solution without the present of kaffir lime essential oil is evaluated at 0.37 which slightly higher than with the present of kaffir lime essential oil. As for consistency coefficient, cassava starch film without the present of kaffir lime have a highest consistency followed with the lowest concentration of kaffir lime essential oil at 0.2% v/v of kaffir lime essential oil.

D. Activation energy of film solution

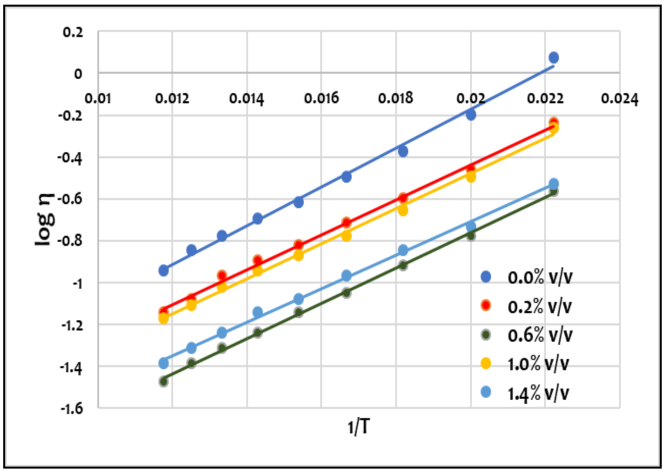

Fig. 4: Linearized Arrhenius equation

Viscosity and temperature was plotted into linearized Arrhenius equation graph as in Figure 4 in order to relate the relationship between temperature and the rheological properties of cassava starch-kaffir lime essential oil. The raw data from rheometer was plotted by following the Arrhenius equation of $\eta = Ae^{-E/RT}$. From the graph, the activation energy of each film solution was obtained. The range of activation energy calculated from the graph were from 662.8420 J/mol to 769.918 J/mol. From the activation energy calculated, 0.0% v/v of kaffir lime essential oil cassava starch solution have the highest activation energy followed by 0.2% v/v, 0.6% v/v, 1.0% v/v and 1.4% v/v. Thus, it can be concluded that the highest viscosity of film solution results in higher activation energy.

CONCLUSION

The rheological properties specifically the viscosity of the cassava film formulation is found to be decreases as the formulation is heated from temperature ranging 45°C to 85°C but does not proportional with the concentration of kaffir lime essential oil in the cassava film solution. The minimum viscosity of 0.23 Pa.s obtained is at temperature 85°C and concentration of kaffir lime essential oil at 0.6% v/v, while the highest viscosity 1.31 Pa.s obtained was at 0.0% v/v of kaffir lime essential oil. The pattern of the shear stress to shear rate graph plotted conclude that this solution was a shear thinning model as the shear stress increases as the shear rate become longer. Meanwhile, the flow behaviour of this solution was determined as pseudoplastic. Also, the activation energy evaluated for the solution is increases as the solution viscosity increases.

RECOMMENDATIONS

Further study on the cassava starch incorporated kaffir lime essential oil can be made by studying other characteristics of this solution. Also, the rheological study can be done varies other component of cassava starch-kaffir lime essential oil film solution such as the amount of cassava starch and glycerol.

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