

Rheological Properties of Cassava Starch Film Forming Solution with Kaffir Lime Oil



Norasmah Mohammed Manshor, Junaidah Jai, Fazlena Hamzah, Noorsuhana Mohd Yusof

Abstract: Biopolymer film has attracted interest in food preservation and packaging. In order to obtain biopolymer film of good characteristics, rheological properties of film forming solution is important. In this study, the rheological properties of cassava starch film forming solution incorporated with kaffir lime oil were determined. The flow behavior measurement was made at shear rate of 0.1 to 100 s^{-1} and 25 °C, whereas the effect of temperature on viscosity was measured at 25 to 70 °C and shear rate of 10 s^{-1} . The flow behavior was well described by power law or Ostwald de Waele model. The film forming solution showed flow behavior index (n) in the range of 0.20 to 0.72 which belonged to a non-Newtonian or pseudoplastic behavior. The shear stress increased with increasing shear rate and temperature. The dependency of temperature on viscosity was described by Arrhenius model which allowed the determination of activation energy (E_a). The E_a of the film forming solutions range between 4.92 to 13.23 $kJ mol^{-1}$. The rheological properties obtained in this study provide a basis to correlate the film forming solution properties to the characteristics of the produced biopolymer film.

Keywords : biopolymer film, film forming solution, flow behavior, rheological properties, viscosity.

I. INTRODUCTION

Food packaging materials from synthetic polymers create a major concern related to health, safety and environmental issue. Synthetic polymers derived from petroleum are non-biodegradable which cause concern over the growing of waste and pollution. The development of new packaging materials from natural and biodegradable sources is an alternative way to reduce the usage of synthetic polymers. Moreover, consumer demand for durable, safe, quality and hygiene food products. Foods are easily deteriorating because of bacteria growth and losses of moisture, gases and nutrition. It becomes spoilage in a short period of time and reduces its

quality. Therefore, antimicrobial film serves as a new approach to food preservation and packaging that may delay or prevent the foods from growth of microorganisms, hence improve quality and extend shelf life of the foods. The film can be consumed with the products without disposal the package and eventhough the film is not consumed, it was anticipated to degrade more readily than synthetic polymer [1].

The process of producing biopolymer film involves preparation of film forming solution which commonly consists of biopolymer, plasticizer and additive or surfactant. Starch is one of the promising materials for film forming solution. Amylose and amylopectin are the main components of starch which have excellent film forming ability to produce good characteristics of film [2]. The incorporation of antimicrobial substances such as kaffir lime oil provide bioactive compound that can improve the functional properties of the produced film. Besides, plasticizer is necessary to improve film's brittleness and increase flexibility and extensibility of the film [3].

In order to produce biopolymer film of excellent barrier and mechanical properties, which in turn depends on the formulation of film forming solution. Different formulation of film forming solution may result in the formation of biopolymer film with different properties. Thus, rheology of the film forming solution is important aspect to consider. The rheological properties describes the flow behavior of film forming solution, hence becomes a vital property because it influences the spreading ability, thickness, uniformity of casting layer and consequently affect the performance of the film [4]. The understanding of the rheological properties of film forming solution is important in the process of casting or coating of film solution onto surfaces of food products by dipping, brushing or spraying [5]. Furthermore, the desired mechanical properties of the produced film rely on the viscoelastic properties of film forming solution.

The flow behavior of the film forming solution can be presented by several models to fitting the shear stress and viscosity as a function of shear rate. The mostly applied model is power law or Ostwald de Waele to determine the flow behavior index (n) and consistency index (k). n is a dimensionless number that indicates a Newtonian fluid when n value is 1, higher than 1 is a dilatant fluid and between 0 and 1 is a non-Newtonian or pseudoplastic fluid. Most of the film forming solution showed pseudoplastic fluid or shear thinning behavior where the shear stress increases with increasing shear rate as reported by Farhoosh & Riazi [6],

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Ghasemlou, Khodaiyan & Oromiehie [3], Abdou & Sorour [7], Kurt & Kahyaoglu [8], Huo et al. [9], and Ma et al. [10]. Although no specific criteria of rheological properties to produce film, the flow behavior of film forming solution allows relationship among characteristics of the obtained film.

The viscosity of film forming solution changes with shear rate, temperature, pressure, moisture and concentration [11]. Peressini et al. [12] have proposed a viscosity lower than 0.7 Pa s for coating applications. However, high viscosity of film forming solution possesses difficulty to remove air bubbles during preparation and obstructs the casting of the solutions [5]. Therefore, using too viscous film forming solution would result a non-uniformity layer of the produced film.

The viscosity of the film forming solution is affected by temperature. For non-Newtonian flow behavior, the viscosity will decrease with increasing temperature. When the temperature increases, the molecular distances and thermal energy of the molecules increases because of acceleration of molecular movement, thus the viscosity decreases [6]. In order to describe the effect of temperature on viscosity, the Arrhenius model was used to determine the activation energy. The activation energy is defined as the energy required to overcome the resistance of fluid to flow and permit the molecular motion [13]. Generally, the activation energy represents the temperature sensitivity of the viscosity. The higher the activation energy, the greater the dependency of temperature on viscosity [6].

Several studies have been done on rheology of film forming solution made of corn starch and sodium alginate [9], chitosan-starch with murta leaf extract [4], cassava starch-konjac glucomannan [14], starch-carrageenan [7], potato starch [15], soy protein isolate [16] and tarra gum [10]. However, rheological properties of cassava starch film forming solution incorporated with kaffir lime oil has not been reported yet. Furthermore, there is limited information on the correlation between the film formulation and the properties of film forming solution which will affect the final characteristics of the produced films. Thus, this work aimed to determine the flow behavior and the temperature variation on viscosity of cassava starch film forming solution incorporated with kaffir lime oil at different film formulations.

II. MATERIALS AND METHODS

A. Materials

Cassava starch (Cap Kapal ABC, Thailand) was purchased from local supermarket in Shah Alam, Selangor. Analytical grade glycerol (Merck, Germany) was used as plasticizer purchased from local supplier. Kaffir lime oil was extracted from kaffir lime peel obtained from local market in Klang, Selangor. The extraction was done by hydrodistillation method using Clevenger apparatus.

B. Experimental Design

A three factor, five level central composite design was chosen to design the experiment using Design Expert 10.0.3. Cassava starch (3-5 % w/v), glycerol (30-50 % w/w starch) and kaffir lime oil (0.2-0.8 % v/v) were used. The formulations for

20 samples are shown in Table- I.

C. Film Forming Solution Preparation

The film forming solutions were prepared according to formulations shown in Table- I. Cassava starch was dissolved in distilled water at 75 °C using hotplate stirrer at 500 rpm for 30 minutes. Then, glycerol was added and the solution was continuously mixed. The film forming solution was cooled to room temperature before kaffir lime oil was added. Next, the film forming solution was stirred further at 500 rpm for 30 minutes to allow thorough mixing.

D. Rheological Properties Measurements

Rheological tests of the film forming solutions were carried out by using electronic rheometer (Physica MCR 300, Anton Paar GmbH, Stuttgart, Germany) with concentric cylinder (CC17) and gap of 0.5 mm. The temperature was regulated by Paar Physica circulating bath and a controlled Peltier system (TEZ 150) with accuracy of ± 0.1 °C. The data of rheological measurements were analyzed using Rheoplus software (v3.40).

The methods were adopted from Ma et al. [10] with modification. The flow behavior of the film forming solutions were measured at 25 °C and shear rate of 0.1 to 100 s⁻¹. The power law model as in (1) was applied to determine the consistency index (k) and flow behavior index (n).

$$\tau = k\gamma^n \quad (1)$$

where τ is shear stress (Pa), γ is shear rate (s⁻¹), k is consistency index (Pa sⁿ) and n is flow behavior index (dimensionless).

The effect of temperature on viscosity of the film forming solutions was measured by temperature sweep test at 25 to 70 °C and shear rate of 10 s⁻¹. Activation energy of viscous flow was determined by (2).

$$\eta = Ae^{\frac{E_a}{RT}} \quad (2)$$

where η is shear viscosity at 10 s⁻¹, A is exponential factor (Pa s), E_a is activation energy (kJ mol⁻¹), R is universal gas constant (J mol⁻¹ K⁻¹) and T is absolute temperature (K).

III. RESULTS AND DISCUSSIONS

A. Flow Behavior of Film Forming Solution

The power law model in (1) was used to determine the flow behavior index (n) and the consistency index (k) from the slope and the intercept of the linear relationship. The model parameters are shown in Table- I with a good coefficient of determination ($R^2 = 0.97-0.99$) indicating the fitness of the power law model to describe the flow behavior of cassava starch-kaffir lime oil film forming solution. The film forming solution shows shear thinning or pseudoplastic behavior by having the values $n < 1.0$. From Table-I, the cassava starch-kaffir lime oil film forming solution shows n values were in the range of 0.20 to 0.72. The degree of pseudoplasticity is higher when the value of n is closer to 0 [17]. For sample SGK 2 and 20, the values were quite high which are 0.60 and 0.72, respectively.

The n values were departed from other samples because the solution contains low concentration of starch which become very dilute solution, thus the degree of pseudoplasticity is low. The n values increased with increasing of cassava starch concentration as depicted by

sample SGK 1, 3, 15 and 17, indicating that the solutions became less pseudoplastic and showing starch concentration dependence on the flow behavior.

The increased of glycerol resulted a decreased of n values for

Table-I: Film forming solution formulations, power law parameters, Arrhenius equation parameters and their coefficient of determination (R²)

Sample	Independent variables			Power law parameters			Arrhenius equations parameters		
	Cassava starch (%w/v)	Glycerol (%w/w)	Kaffir lime oil (%v/v)	n	k (Pa s ⁿ)	R ²	E _a (kJ mol ⁻¹)	A x 10 ⁻² (Pa s)	R ²
SGK 1	3	30	0.2	0.24	2.70	0.99	5.88	3.88	0.99
SGK 2	3	30	0.8	0.60	0.10	0.99	10.57	0.12	0.99
SGK 3	3	50	0.2	0.20	3.33	0.99	6.47	4.14	0.99
SGK 4	3	50	0.8	0.24	3.05	0.99	7.40	2.88	0.99
SGK 5	4	40	0	0.32	8.50	0.99	4.92	27.65	0.97
SGK 6	4	40	0.5	0.27	4.62	0.99	7.33	6.66	0.99
SGK 7	4	40	0.5	0.27	4.01	0.99	8.07	3.79	0.97
SGK 8	4	40	0.5	0.30	3.88	0.99	6.82	8.98	0.97
SGK 9	4	40	0.5	0.31	3.57	0.99	7.16	9.08	0.99
SGK 10	4	40	0.5	0.26	6.16	0.99	6.29	19.56	0.99
SGK 11	4	40	0.5	0.29	5.22	0.99	5.50	18.48	0.98
SGK 12	4	40	1.0	0.31	9.11	0.99	5.13	23.90	0.97
SGK 13	4	23.2	0.5	0.33	6.30	0.98	13.23	0.74	0.99
SGK 14	4	56.8	0.5	0.32	4.22	0.99	6.86	6.41	0.99
SGK 15	5	30	0.2	0.38	11.49	0.99	12.78	2.53	0.99
SGK 16	5	30	0.8	0.27	14.68	0.99	6.50	16.79	0.97
SGK 17	5	50	0.2	0.31	12.27	0.99	6.05	25.57	0.99
SGK 18	5	50	0.8	0.22	8.00	0.97	8.62	3.69	0.99
SGK 19	5.7	40	0.5	0.37	12.77	0.99	5.44	52.82	0.99
SGK 20	2.3	40	0.5	0.72	0.02	0.99	9.40	0.08	0.99

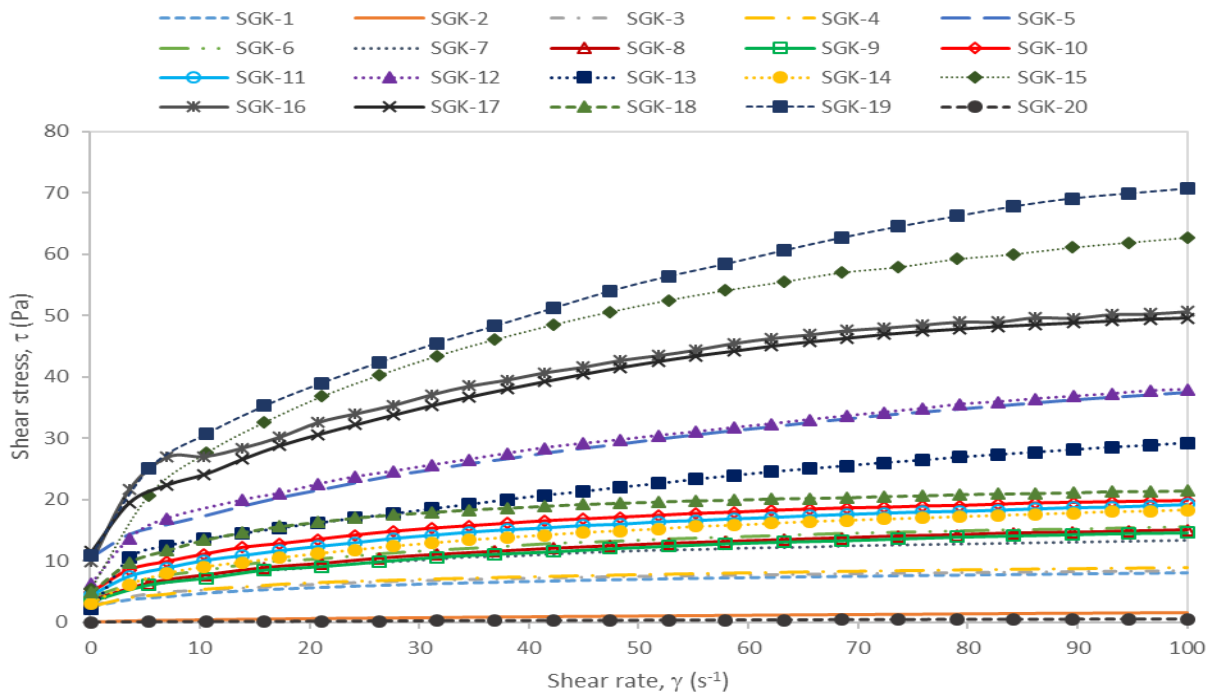


Fig. 1. Shear stress vs. shear rate for cassava starch-kaffir lime oil film forming solution

all samples. The increased of kaffir lime oil also decreased the n values except for sample SGK 1, 2, 3 and 4. The similar shear thinning behavior of other starch solution was also reported by Bertuzzi, Armada, & Gottifredi [18]; Chillo et al. [19]; Choi & Yoo [20] & Kim & Yoo [21].

The shear thinning behavior also indicated by increasing of shear stress with shear rate. All samples had the same behavior as shown in Fig. 1. The shear thinning behavior can be described by a starch network entanglement mechanism during shearing. Increasing shear rate gradually results in disentanglement of the arrangement of long chain molecules and consequently less intermolecular resistance to flow [22]. The samples with high starch concentration have higher shear stress, but glycerol and kaffir lime oil addition did not affect the shear stress as the shear rate increasing, as depicted from Fig. 1. This is in agreement with Chen, Kuo, & Lai [23] who reported that the use of glycerol in tapioca starch/decolorized hsian-tsoo gum film solutions did not have a strong effect on the rheological properties because glycerol is a quite small molecule as compared to tapioca starch and hsian-tsoo gum. From Fig. 1, sample SGK 2 and 20 had the lowest shear stress as compared to other samples. This was synchronized with the high n values obtained which show less pseudoplasticity behavior of the samples.

The consistency index (k) found from the power law model presented the viscous nature of the film forming solution. The k values increased with increasing starch concentration for all samples as shown in Table- I. At 40 %w/w glycerol and 0.5 %v/v kaffir lime oil, the increased of k values is in line with the decreasing of n values with increasing of starch content. This show the film forming

solution was more viscous as the starch concentration was increased. This viscous nature may be resulted by an associative effect of hydrophobic chains in starch [24].

The relationship of viscosity and shear rate can be observed from Fig. 2. The viscosity of the film forming solutions decreased with increasing shear rate which confirmed the indication of shear thinning behavior ($n < 1.0$) [8]. Because the solution is pseudoplastic, the aggregated particles disrupted during shearing causes less resistance to flow and the viscosity decreases until nearly a constant value [7]. All samples show the same trend of decreasing viscosity with increasing shear rate, regardless of the concentration of glycerol and kaffir lime oil as shown in Fig. 2. The independent effect of glycerol on the viscosity of film forming solution can be caused by the types and concentrations of film forming materials and also the solvent used for film forming solution [23].

At shear rate from 0.1 to 100 s^{-1} , the viscosity of the film forming solutions range from 0.015 to 6.090 Pa s . The higher the concentration of starch, the higher the viscosity. For large scale application, the process of producing film mostly involve uniform mixing, pumping of solutions through filters, transfer to the film casting line and spreading the solution smoothly, for which a suitable viscosity is $1-10 \text{ Pa s}$ [25]. As stated by Wu et al. [26], non-uniform films are resulted from high or less viscous film forming solutions, hence moderate viscosity is required to form film of good characteristics. The higher the shear rate, the lower the viscosity. Therefore, in this study the viscosity obtained by most samples at shear rate of 10 s^{-1} are within suitable range for film casting applications.

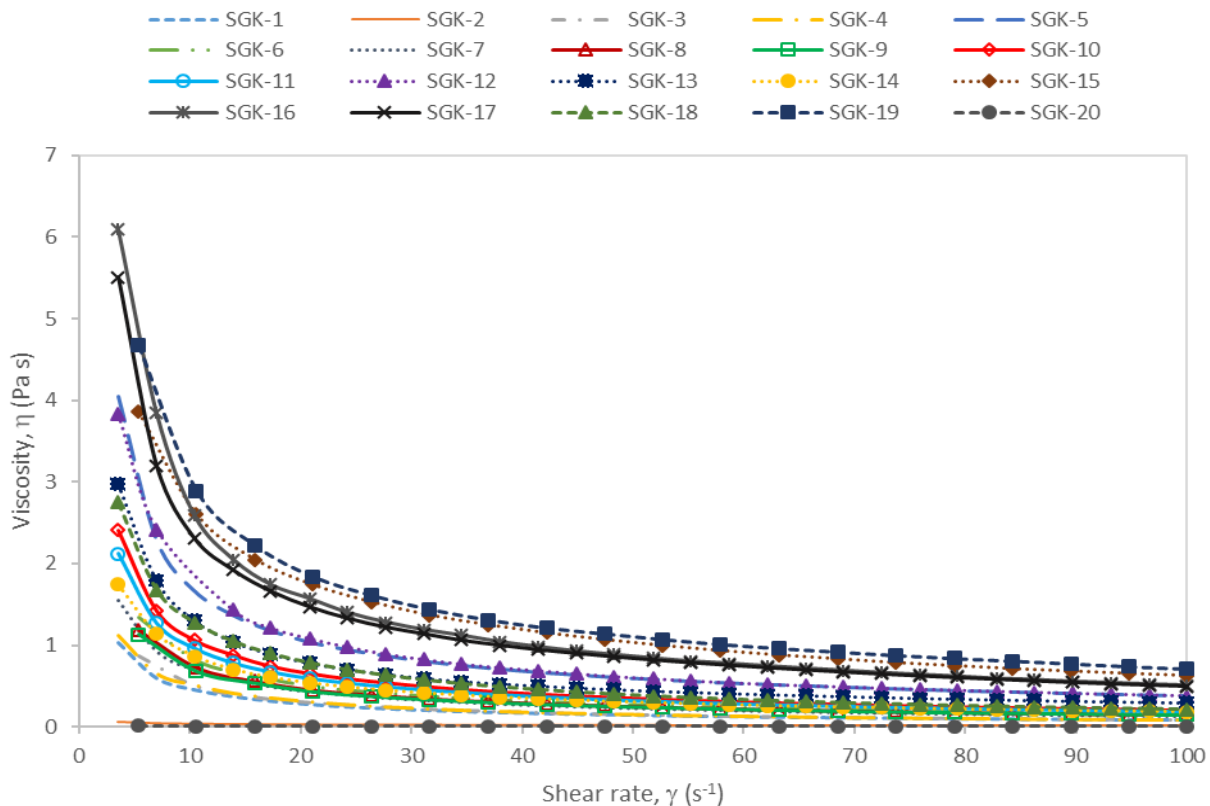


Fig.2. Viscosity vs. shear rate for cassava starch-kaffir lime oil film forming solution

B. Temperature Dependency on Viscosity

At shear rate of 10 s^{-1} , the viscosity for all samples decreased as the temperature increases from 25 to $70 \text{ }^\circ\text{C}$ as shown in Fig. 3. This behavior was similar to other film solution of pullulan-sodium alginate [27], salep glucomannan [8], high amylose corn starch [18] and sweet potato starch-xanthan gum [20]. The increasing of temperature attributed in increased of intermolecular distances as a result of thermal expansion, hence the viscosity decreased [28].

The temperature dependency on viscosity for cassava starch-kaffir lime oil film forming solution was well described by Arrhenius equation in (2) with R^2 of 0.97-0.99 as summarized in Table- I. The activation energy (E_a) and exponential factor (A) were obtained from the slope and intercept of the linearized Arrhenius equation. The E_a values were in the range of 4.92 to $13.23 \text{ kJ mol}^{-1}$. These values were not far from E_a of other film forming solution which are 22.51 to $24.85 \text{ kJ mol}^{-1}$ for pullulan-sodium alginate [27], 18.823 to $22.248 \text{ kJ mol}^{-1}$ for high amylose corn starch [18], 8.29 to $18.10 \text{ kJ mol}^{-1}$ for sweet potato starch-xanthan gum [20] and 16.5 to 19.0 kJ mol^{-1} for acorn starch [21].

The activation energy of viscous flow is defined as the minimum energy required to overcome the resistance to flow allow molecular motion to occur [9]. The higher E_a shows the more dependence of temperature to viscosity of the solution [16]. The lower E_a values may be related to lower flow

behavior index (n) as found by Choi & Yoo [20] in sweet potato starch-xanthan gum mixtures. It means that, the more pseudoplastic the solutions (n closer to 0), the lower the effect of temperature on viscosity. From Table- I, this similar relationship of n and E_a is shown by most samples. The study by Bortnowska, Krzemińska, & Mojka [29] reported that increase in starch content decreases the E_a and decreases the temperature dependency of starch suspension. This similar trend was observed for all samples except SGK 1, 4, 15 and 18 as shown in Table- I.

IV. CONCLUSION

The rheological properties of cassava starch film forming solution with kaffir lime oil were reported in this study. The film forming solution exhibited a shear thinning or non-Newtonian behavior indicated by increasing of shear stress with shear rate. The flow behavior index (n) of the film forming solution obtained from power law equation was less than one. The viscosity of the film forming solution was in acceptable range to form biopolymer film. From Arrhenius equation, the higher activation energy (E_a) shows the higher effect of temperature on viscosity. The rheological properties of cassava starch-kaffir lime oil film forming solution found in this work provide a basis to relate with characterization of the produced biopolymer film.

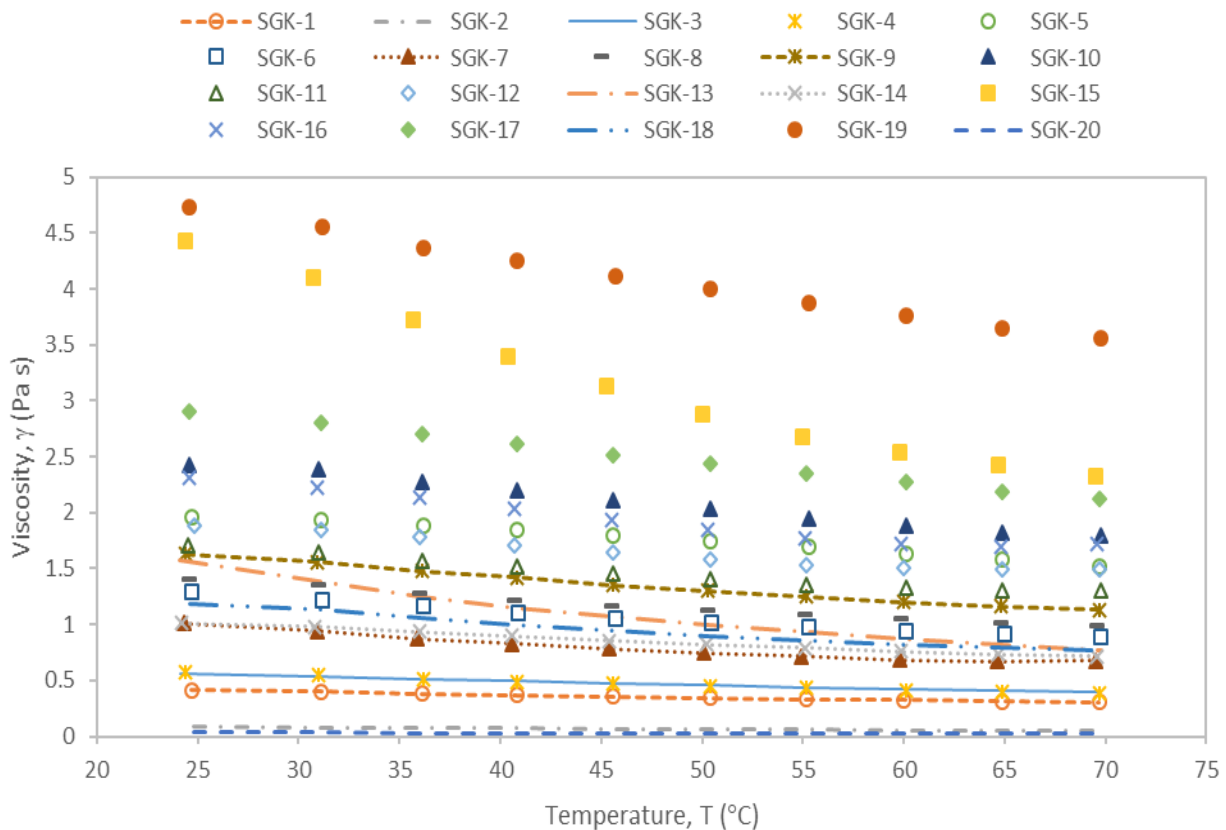


Fig. 3. Viscosity vs. temperature of cassava starch-kaffir lime oil film forming solution

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