Rheological characterization and modelling of glucose syrup production process from selected agricultural crops

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Abstract: Rheological characterization and development of models for glucose syrup production process obtained from selected agricultural crops were studied. Glucose syrups were produced at 60°C, 80°C and 100°C. The rheological properties of the glucose syrups were investigated and the effect of temperature on viscosity was determined. Also, the effect of shear rate on viscosity was determined at spindle speeds of 10, 20, 50 and 100 rpm. Data obtained were fitted into Power Law model for analyzing the viscosity and for fluid classification. The starch yields were 71.7%, 85.3% and 91.5% and the syrup yields were 78.82%, 86.35% and 88.65% for maize, rice and cassava respectively. Viscosities of the syrups showed an inverse relationship with temperature and shear rates. The Power Law index (*n*) was found to be between 0 and 1 and the Power Law model plots showed distinct characteristics of pseudo-plastic behaviour under the classification of non-Newtonian fluid.

Keywords: Glucose, Power Law model, viscosity, pseudo-plastic, Non-Newtonian fluid

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1 Introduction

Glucose syrup is food syrup made from the starch of carbohydrate crops such as maize, rice, cassava, millet, sorghum amongst many others and composed of mainly glucose. It is the most valuable substitute for sugar, and also it is an aqueous solution of several compounds, principally glucose, dextrose, and maltose. It is a purified concentrated solution of nutritive monosaccharide and higher saccharine (Ayernor et al., 2002). Glucose syrup is mainly used in commercially prepared food such as confectionaries (e.g. chewing gums and chocolates), jams, jellies and canned fruits, pharmaceuticals (e.g. cough and vitamin based tonics, cough lozenges, and as a granulating agent for tablet coating). It is also used in the production of leather products and bakery products as thickener and sweetener as well as preservatives in gumming properties in traditional oil extraction industries amongst others. Glucose syrup is made from the hydrolysis of starch which can be acid hydrolysis, enzyme hydrolysis, or the combination of the two (Barnes et al., 1989). Enzymatic hydrolysis process produces higher yields of desired products and lesser formation of unwanted products (Sanjust et al., 2004). This makes the product superior to the one produced by the conventional acid hydrolysis process. Irrespective of the feedstock or the method used for hydrolysis, certain steps are common to the production of glucose syrup which include starch separation from the plant material, and starch gelatinization (by heating the ground, cleaned feedstock) which causes the breakdown of the intermolecular bonds of the starch molecules, allowing the hydrogen bonding sites to engage more water. Thereafter, glucose syrup can be produced by either acid hydrolysis or enzyme hydrolysis method.

Rheology, defined as the science of material flow and deformation, is a fundamental interdisciplinary science gaining increasing importance in the field of foods. It is used to characterize flow behavior of foods and other materials as well as the structural characteristics.

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Consequently, basic rheological information on materials is important not only to engineers, but also to food scientists, processors, and others who may utilize this information and find new applications. One important rheological property of food materials is viscosity (Rao and Anantheswaran, 1982). Fluids are classified as either Newtonian or non-Newtonian fluids. The fluids which obey Newton's law of fluid flow are called Newtonian fluids (Schramm, 1994). A Newtonian liquid starts to flow when a stress is supplied, and deformation stops instantly when the stress is removed (Morrison, 2001). In Newtonian liquids, the viscosity is constant with respect to time of shearing and it does not change in the re-testing situation (Barnes et al., 1989). Non-Newtonian fluids are those which do not obey Newton's law of fluid flow. The relationship between shear stress and shear rate is not constant. There are several types of non-Newtonian flow behaviour, characterized by the way a fluid changes in response to variations in shear rate. The most common types of non-Newtonian fluids are: pseudo plastic, dilatant plastic, thixotropy and rheopexy. Liquid foods that contain relatively small molecules exhibit Newtonian behaviour, while those containing dissolved polymers, insoluble solid or immiscible fluids exhibit non-Newtonian flow behaviour. In characterizing the fluids, several models can be used which include the Power Law model, Bingham plastic model, Herschel-Bulkley model, modified Casson model amongst others. The Power Law model is an easy-to-use model that is ideal for shear-thinning, relatively mobile fluids such as weak gels and low-viscosity dispersions. Its simplicity in describing fluid behaviour, permitting mathematical predictions, and correlating experimental data give it advantage over several other models. This research therefore studied the rheological characterization of glucose syrup produced from cassava, maize and rice using the Power Law model; and modelled the glucose syrup production process.

2 Materials and methods

2.1 Sample preparation

Maize (white), rice and cassava were purchased, inspected, cleaned and washed to remove impurities like stones, cobs, dust particles and foreign materials after which they were ready for steeping. Cleaned samples weighing 5 kg each of the raw materials (cassava, rice and maize) were steeped separately in water at ambient temperature of 32°C for 72 h hours to soften the materials for easy grinding and starch extraction. The samples (maize, cassava and rice) were separately ground to fine particles. The grinding was conducted with care and several washing of the machine to avoid contamination with foreign materials. This operation was followed by mashing the ground materials to remove and break lumps that could affect starch extraction. Starch extraction was carried out by passing the slurry several times through a perforated sieve to remove further impurities, and the slurry which contained pure starch was allowed for 24 hours to sediment. After sedimentation, the top water was carefully poured off, and the final slurry (thick slurry) was squeezed using a muslin cloth. The starches were exposed to sunlight for drying to obtain dry particles. The dry matter recovered from the starch was determined using Equation (1) as adopted by Ji et al. (2004) as:

$$\% \text{ Yield} = \frac{\text{Dry weight of starch recovered from extraction}}{\text{Dry weight of whole sample taken}} \times 100 \quad (1)$$

A starch solution was made (30%-40% solution w/w) from distilled water as recommended by Pelembe et al. (2002). The solution was gelatinized by heating (process of breaking down the intermolecular bonds of starch molecules in the presence of water and heat).

2.2 Hydrolysis of the gelatinization

This was done in three stages viz.

2.2.1 Liquefaction of the starch (using alpha-amylase)

This is the first stage. After gelatinization, the solution became very viscous and was cooled to room temperature. The pH was adjusted with phosphate buffer solution to 4.5, followed by the introduction of α-amylase and was stirred for approximately 2 h. This was done to liquefy the starch solution by breaking down the α-1, 4-glycosidic bond to yield molecules of branch glucose as proposed by Wiseman (1987).

2.2.2 Saccharification using Glucoamylase (γ -amylase)

This is the second stage. It involved the introduction of γ-amylase (pH 4.6) into the liquefied-starch solution and was stirred for approximately 4 h. Afterwards, the temperature was raised to 55°C and stirred for approximately 25 min. The solution was then allowed to cool (crude glucose syrup).

2.2.3 Clarification

This is the third stage. After hydrolysis, the syrup (crude glucose syrup) was passed through sieve to remove impurities resulting from starch particles that were unbroken down and also to improve its colour and stability.

2.3 Viscosity

The viscosity of glucose syrup was determined using Fann Viscometer (Model 35A Fann Viscometer, Houston, Texas, USA). The viscosities of the whole samples of different syrups were determined at 30, 40, 50 and 60°C using a stove and water bath with a thermometer to determine the effect of temperature on viscosity in order to classify the fluids. Appropriate spindle was used to determine the viscosity depending on the thickness of the sample as described by Nkama (1993). The reading was measured at four different shear rates, 10, 20, 50, and 100 rpm. The results obtained were multiplied by appropriate factor based on the spindle used to obtain the viscosity in centipoises unit and it was divided by 1000 to obtain the viscosity in N sm^{-2}.

All experiments were conducted in triplicates and average values were recorded.

2.4 Fitting of data to models

The data obtained were fitted to the Power Law model which was used for analyzing the viscosity and fluid classification. The Power Law model is given as:

$$\mu_{app} = K\gamma^{n-1} \quad (2)$$

where, K = consistency index; n = power law index; μ_{app} = apparent viscosity (N sm^{-2}); γ = Shear rate (rpm).

On linearization, the equation becomes,

$$\ln\mu_{app} = \ln K + (n-1)\ln\gamma \quad (3)$$

A plot of of $\ln\mu_{app}$ against $\ln\gamma$ is used in obtaining the Power Law and consistency index.

For Newtonian fluids, $n = 1$; for pseudo-plastic fluids, $0<n< 1$; and for dialatent materials, $n>1$ (Finney, 1973).

2.4 Statistical Analysis

The data obtained from the experiments were statistically analyzed using SPSS software package version 20.0 and Minitab software package version 16.0.

3 Results and discussion

3.1 Starch and syrup yield

The starch yields obtained for maize, rice and cassava were 71.7%, 85.3% and 91.5% respectively. Comparatively, cassava was found to have the highest starch yield, and maize had the lowest. The result obtained was similar to the findings of Omemu et al. (2004) which observed the highest starch yield from cassava and the lowest from maize. This implies that starch yield is dependent on crop type. The syrup yields obtained were 78.82%, 86.35% and 88.65% for rice, maize and cassava respectively. This was highly dependent on the percentage starch yield. This was in line with the findings of Zainab et al. (2011) which observed similar trend while working on maize, millet and sorghum. Figure 1 shows the starch and syrup yield for the three crops, while Figure 2 shows the effect of temperature on syrup yield on rice, maize and cassava. It was found that syrup yield was highly affected by raw material type and temperature. Temperature is required to breakdown the starch particles to enable fast enzymatic reaction. The conversion process was faster at higher temperature. This was in agreement with the findings of Alias (2009) which observed highest increase in glucose yield percentage as temperature rises.

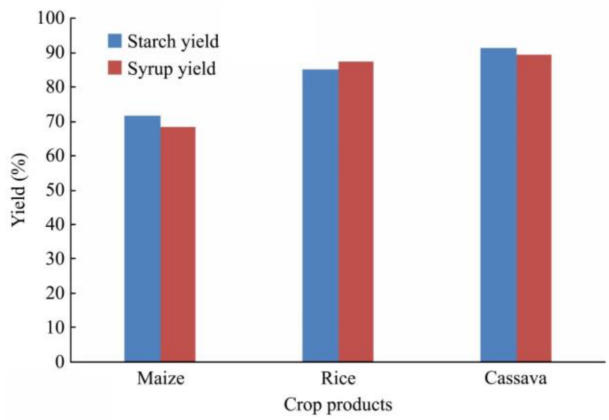

Figure 1 Starch and syrup yields for the crop products

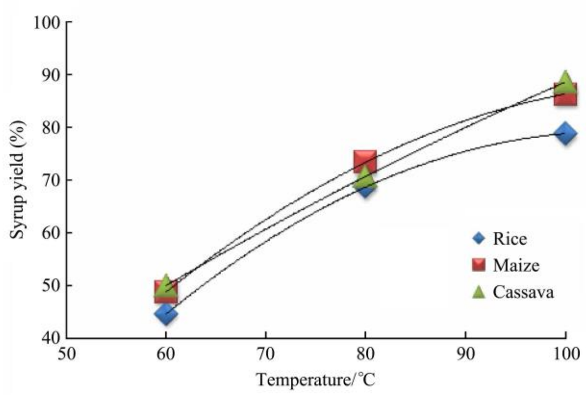

Figure 2 Effect of temperature on syrup yield for the crop products

3.2 Effect of temperature on viscosity

It was observed that viscosity decreased for all the three crop types with increase temperature (Figure 3). An activation energy is necessary for moving of a molecule, and as the temperature increased, the liquid flowed more easily due to higher activation energy, thereby decreasing the resistance to gradual deformation by shear stress. This observation was in agreement with earlier findings by Finney (1973), Rha (1978), Heikal and Chhinnan (1990) and Sengul et al. (2005). The effect of temperature on viscosity was used to characterize the fluids as non-Newtonian on the basis of their flowability. The classification was validated by the effect of shear rate on viscosity.

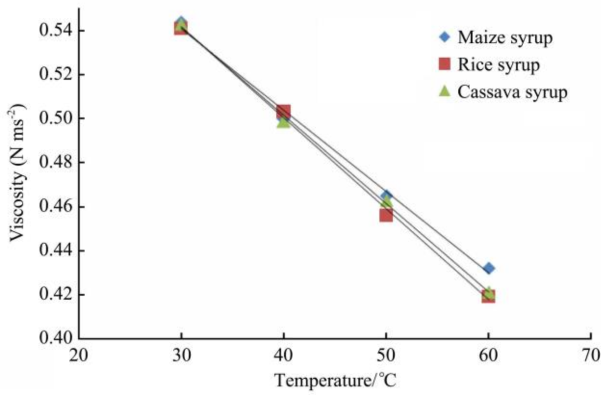

Figure 3 Effect of temperature on viscosity of the syrups

3.3 Effect of shear rate on viscosity

The effect of shear rate on viscosity for maize, rice and cassava syrups is shown in Figure 4. Viscosity in all the cases was found to decrease with increased shear rate. The relationship between viscosity and shear rate from the viscometer for all syrup samples suggested that glucose syrup is generally a shear thinning of the pseudo-plastic fluid material type as compared with the characteristic curve of shear thinning food materials. This confirmed earlier literatures that other classes (dialatent, rheopectic and thixotropic) of non-Newtonian behaviour are relatively rare in food systems.

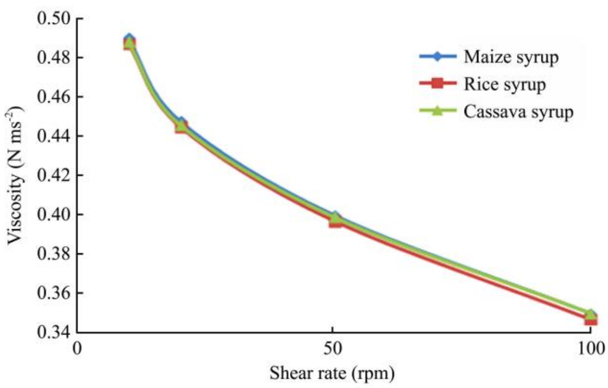

Figure 4 Effect of shear rate on viscosity of the syrups

3.4 Characterization of the glucose syrup using the Power Law model

The viscosity and shear data obtained were utilized in calculating shear stress and the values were fitted to Power law model. The Power Law model is normally used on data that show monotonically shear thinning behaviour, rather than on data areas that show plateaus, that is, the zero shear and infinite shear viscosities. A plot of of lnμ_{app} against lnγ in Figures 5-7 for maize, rice and cassava respectively gave a straight line graph with slope $(n-1)$ and an intercept K. The negative slope of straight line shows the non-Newtonian/shear-thinning nature of the syrups. Similar result of negative slope was reported for mango juice and tomato concentrate by Dak *et al.* (2006) and Dak *et al.* (2008) respectively. The slope and intercept values were used to calculate the flow behavior/Power Law index and consistency index presented in Table 1; which were used to describe the rheological behavior of the different food materials as opined by Sopade and Kassum (1992). The Power Law index (n) was found to be between 0 and 1 (Table 1) which showed that the samples are shear thinning and revealed distinct characteristics of pseudo-plastic behaviour under the classification of non-Newtonian fluid.

Table 1 Slope, intercept, flow behaviour index and consistency index for the model

Sample	Temp. (ºC)	Slope, m	Intercept, c	Flow Behaviour Index, n	Consistency Index, k
Maize	30	–0.1079	–0.2405	0.8931	0.7862
	40	–0.1293	–0.2531	0.8707	0.7764
	50	–0.1284	–0.3298	0.8716	0.7191
	60	–0.1431	–0.3765	0.8569	0.6863
Rice	30	–0.1126	–0.2303	0.8874	0.7943
	40	–0.2431	–0.2431	0.8652	0.7842
	50	–0.1348	–0.3260	0.8681	0.7218
	60	–0.1441	–0.3801	0.8559	0.6838
Cassava	30	–0.1091	–0.2389	0.8909	0.7875
	40	–0.1308	–0.2389	0.8694	1.2863
	50	–0.1300	–0.3287	0.8588	0.7199
	60	–0.1412	–0.3856	0.8588	0.7199

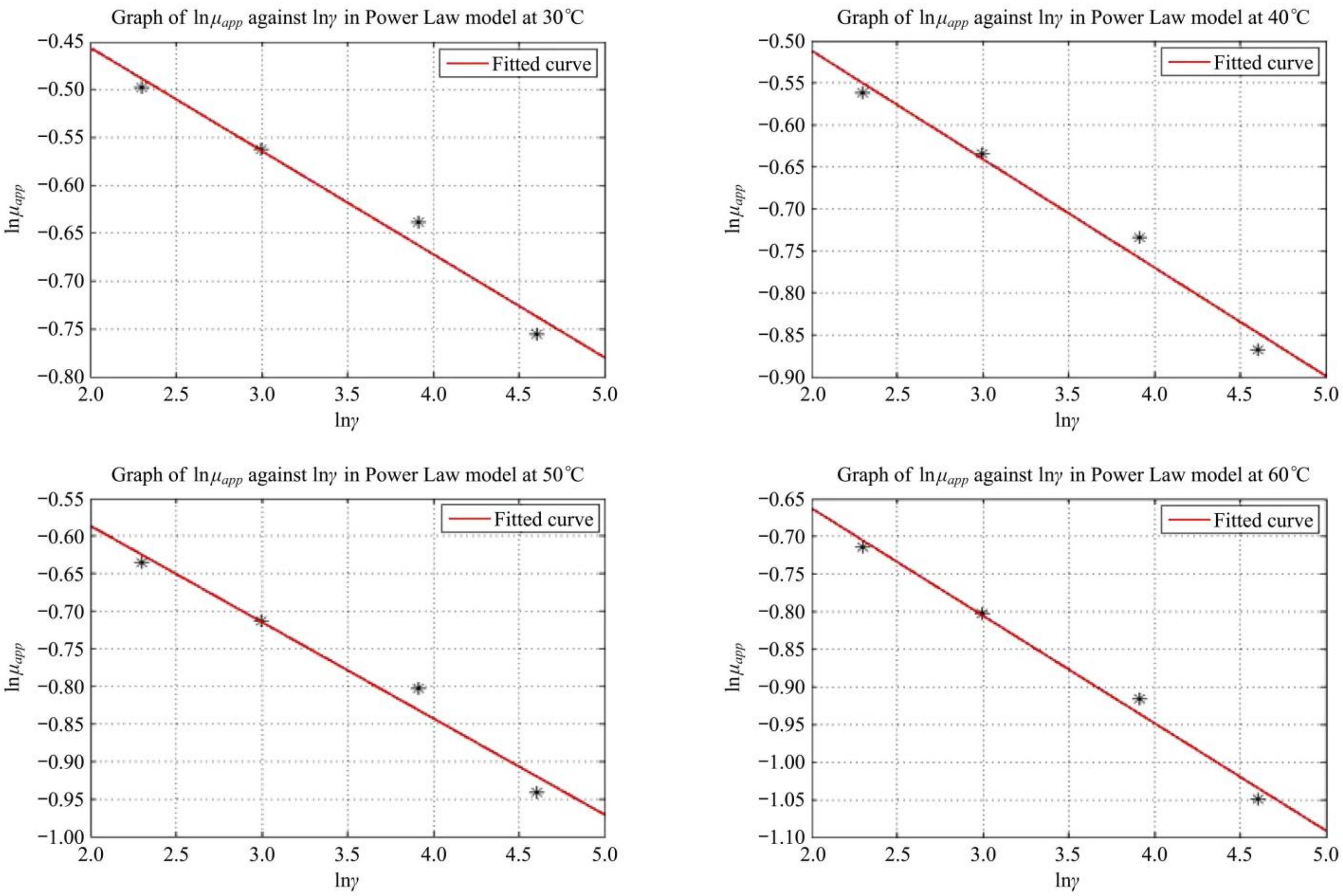

Figure 5 Power Law model for maize

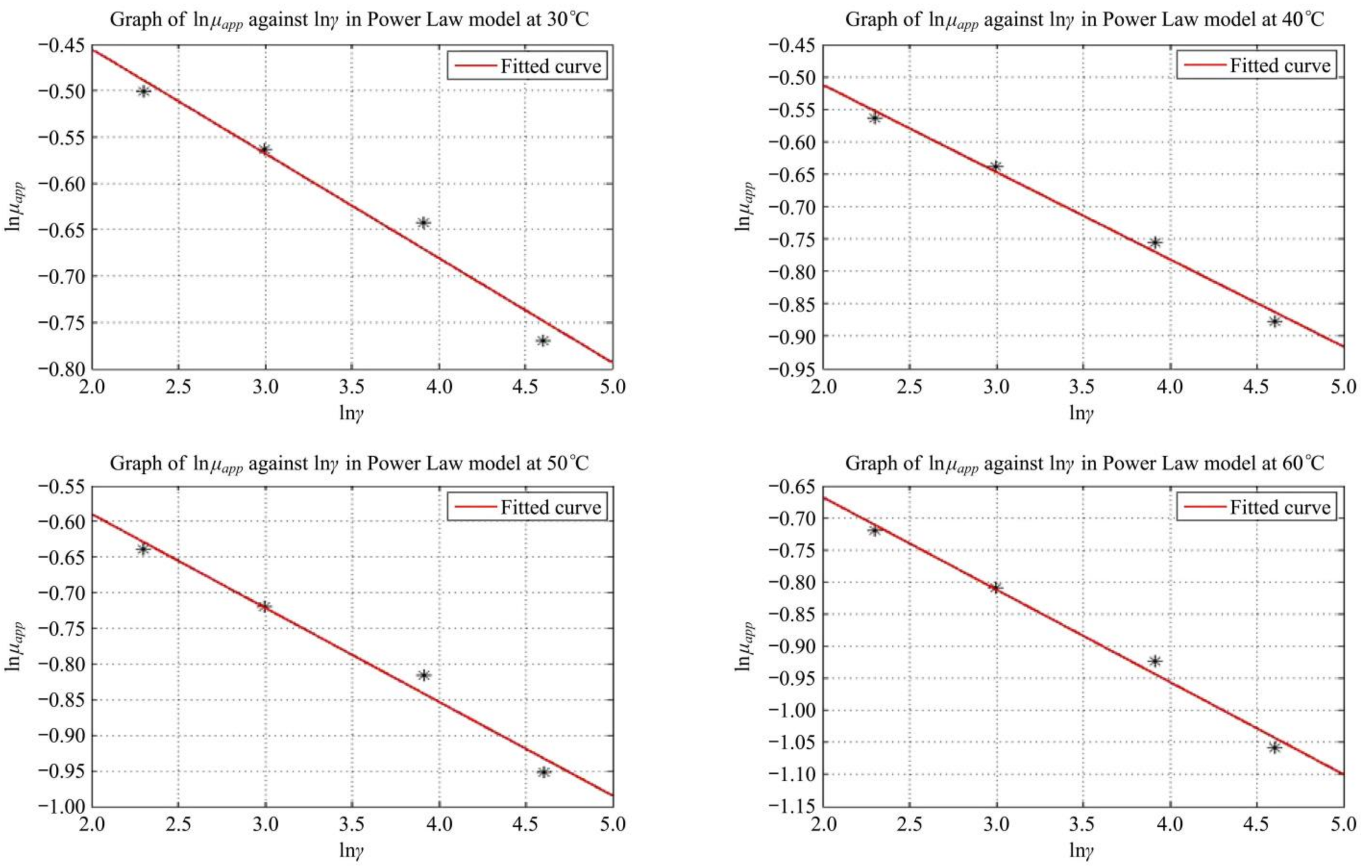

Figure 6 Power Law model for rice

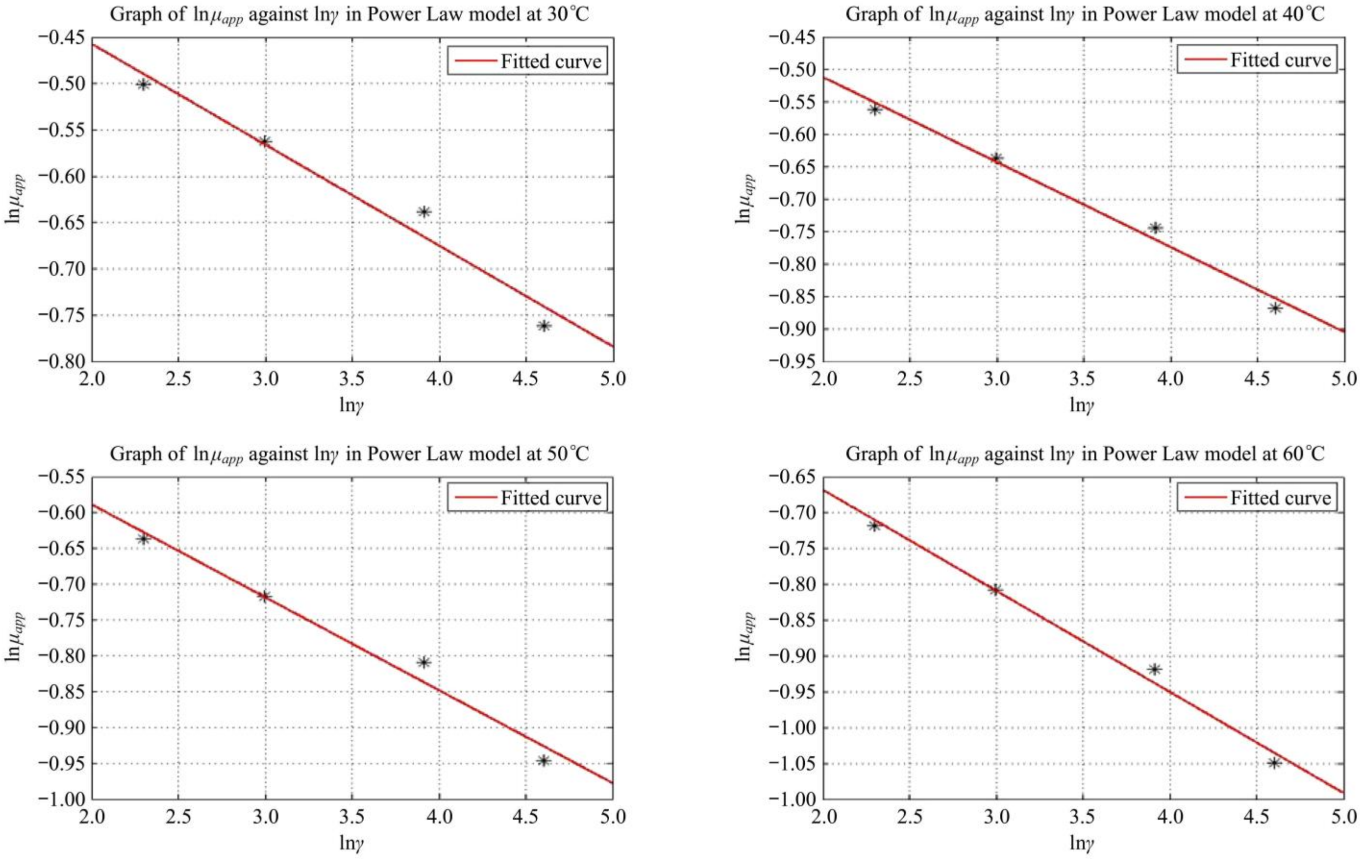

Figure 7 Power Law model for cassava

3.5 Analysis and Modelling of the viscosity

Table 2 shows the test of between-subjects effect of temperature and shear rates on viscosity which is the dependent variable. It was observed that both shear rates and temperatures have significant effect on the viscosities of sample, whereas, the sample type has no significant effect. Figure 8 shows the interaction plot for viscosity for all the samples. Equations (4-6) show the model equations for the viscosities of maize, rice and cassava respectively.Though it has been well established that temperature has a strong influence on the viscosity of fluids, with viscosity generally decreasing with increase in temperature (Rao, 1999; Diamante and Lan, 2014), however, these generated models showed the importance of the shear rate dependence of viscosity. Therefore, viscosity must be specified at a given rate of shear and temperature. This proposition was equally echoed by Ukwuoma and Ademodi (1999).

$$VS_{Maize} = -0.72+0.12SR_{10}+0.07SR_{20}-0.05SR_{50}-0.14SR_{100}+0.11T_{30}+0.06T_{40}+0.05T_{50}-0.12T_{60} \quad (4)$$

$$VS_{Rice} = -0.75+0.15SR_{10}+0.06SR_{20}-0.05SR_{50}-0.17SR_{100}+0.13T_{30}+0.05T_{40}-0.05T_{50}-0.13T_{60} \quad (5)$$

$$VS_{Cassava} = -0.74+0.14SR_{10}+0.06SR_{20}-0.04SR_{50}-0.16SR_{100}+0.13T_{30}+0.04T_{40}+0.04T_{50}-0.12T_{60} \quad (6)$$

Table 2 Tests of between-subjects effects

Dependent Variable: Viscosity

Source	Sum of squares	df	Mean square	F	Sig.
Corrected Model	1.038	8	0.130	174.834	<0.0001[a]
Intercept	26.119	1	26.119	35204.510	<0.0001[a]
Sample	0.004	2	0.002	2.756	0.076
Shear Rate	0.605	3	0.202	271.716	<0.0001[a]
Temperature	0.429	3	0.143	192.672	<0.0001[a]
Error	0.029	39	0.001	-	-
Total	27.19	48	-	-	-
Corrected Total	1.07	47	-	-	-

Note: [a] = Significant.

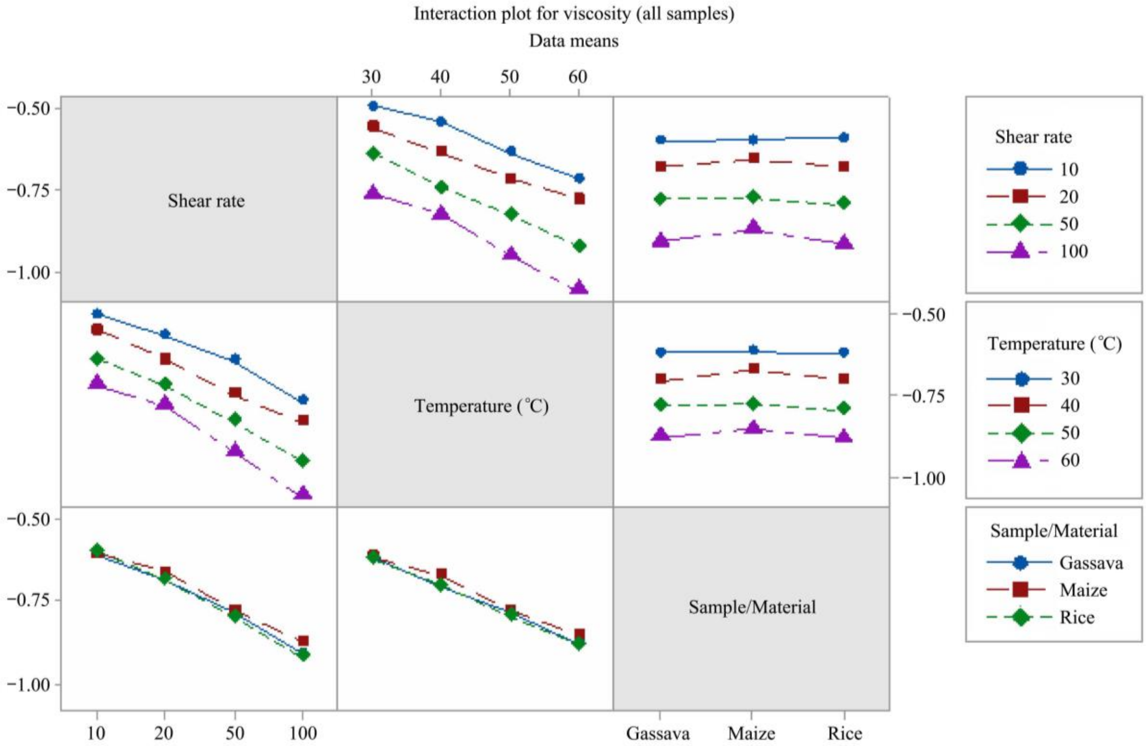

Figure 8 Interaction plot for viscosity

4 Conclusions

Glucose syrups were produced from selected agricultural crops viz maize, rice and cassava. It was observed that cassava had the highest starch and syrup yields compared to maize and rice. Viscosities of the syrups showed an inverse relationship with temperature and shear rate, hence characterizing them as non-Newtonian fluids. The data obtained were fitted into the Power Law model. The Power Law index (*n*) was found to be between 0 and 1 and the Power Law model plots showed distinct characteristics of pseudo-plastic behavior under the classification of non-Newtonian fluid.

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