Flow behaviour and gelatinization kinetics of *Brachystegia eurycoma* flour and starch dispersions

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Abstract: *Brachystegia eurycoma* flour (5%) and starch (2.6%) dispersions heated for 2,5,10,15,30 and 44 min at 75 ℃, 80 ℃, 85 ℃ and 90 ℃ were characterized by pseudoplastic behaviour ($0.203<n<0.329$) and ($0.249<n<0.429$) respectively with shear thinning effect, and increasing shear stress with shear rate. The consistency coefficient, *k*, increased with increasing heating time, while the flow behaviour index, *n*, decreased with increasing heating time. Maximum viscosities attained due to heat-induced gelatinization showed a power relationship with heating temperature. The viscometric measurements showed that starch gelatinization followed a first order kinetic model with the rate constants showing an Arrhenius temperature dependence with an activation energy of 143.43 and 120.91 kJmol^{-1} for *Brachystegia eurycoma* starch and *Brachystegia eurycoma* flour.

Keywords: flow behavior, gelatinization kinetics, activation energy, *Brachystegia eurycoma*

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1 Introduction

Brachystegia eurycoma, called 'achi' in Igbo speaking states of Nigeria is a food gum. The seeds can be used for food and feed, since they are a good source of starch and protein with 48.2% and 12.4% respectively (Ikegwu et al., 2009). Starches have been used since ancient times as raw material to prepare different products. They are employed in foods because of their good thickening and gelling properties.

An important quality index of *Brachystegia eurycoma* is its thickening quality. In quantitative terms, thickness can be measured in terms of consistency index or viscosity. The knowledge of the rheological properties is important for design and evaluation of the process, process control (Brennan et al., 1980; Dail and Steffe, 1990a, 1990b), consumer acceptability of a product, (Harrison and Cunningham, 1985) and the optimization of process variables. The understanding of the rheological characteristics of *Brachystegia eurycoma* starch at different temperatures is crucial for effective design and simulation of its momentum transfer process and system. The effects of transport and manipulation upon the physical integrity of fresh or cooked foods, their behaviour during the processes of elaboration and texture quality of the product depend on their answer when external forces are applied (Aguilera, 1997), but also shear rate and time of shearing (Tiu and Boger, 1974). Almost all food is heat treated before being eaten. During the heating process a lot of different physical, chemical, and structural changes take place (Thorvaldson et al., 1999), for example, diffusion of heat and water, gelatinization of starch, denaturalization of proteins, formation of aroma compounds, and so on. Okechukwu et al. (1991) reported that starch rigidity onset takes place at temperature above 70 ℃.

The power law rheological model has been used to describe the flow behaviour of gelatinized starch suspensions (Evans and Haisman, 1979; Doublier, 1981; Kubota et al., 1979; Bagley and Christianson, 1982; Okechukwu and Rao, 1996).

Several studies have reported on gelatinization kinetics

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of starches (Bakshi and Singh, 1980; Kubota et al., 1979; Lin, 1993; Ramesh, 2001; Suzuki et al., 1976; Zhong and Sun, 2005). In comparison, there is limited information in the literature regarding the flow behaviour of gelatinized *Brachystegia eurycoma* starch dispersion. The present investigation aims to estimate kinetic parameters of *Brachystegia eurycoma* flour and starch dispersions. The data generated from this study would be a useful indicator in the design and control of thermal processing of foods.

2 Materials and methods

2.1 Sample preparation

The *Brachystegia eurycoma* seeds used in this study were collected from ‘achi’ Ejum River Izzi in Abakaliki, Ebonyi State, Nigeria. The choice of site is for uniformity. The flour and starch were extracted from the *Brachystegia eurycoma* seeds according to the method of Ikegwu et al. (2010).

2.2 Gelatinization kinetics

A procedure based on the methods of Kubo*ta et al.* (1979) as modified by Okechukwu et al. (1991) was used for gelatinization kinetics of starch in *Brachystegia eurycoma* flour. About 20g of *Brachystegia eurycoma* flour was thoroughly mixed with 49 mL of distilled water at 50 ℃ in a 500 mL beaker. 331 mL of hot water at a pre-calculated temperature was added to the 40 ℃ dispersion. The water was just enough to produce 5% hot paste instantaneously raised the paste temperature to that selected for investigation and to cover the groove of the viscometer spindle. After a brisk stirring not exceeding 30 s, the hot suspension was quickly poured into a stainless steel vessel and agitated with a magnetic stirrer at the selected temperature, which was maintained over a stirrer hot plate. At specific time intervals, samples of hot paste were withdrawn and poured into another 500 mL beaker, immersed in an ice bath, and cooled rapidly. The viscosity of the cold gelatinized paste was then measured using the NDJ-85 digital display viscometer. A similar procedure was adopted for the starch gelatinization: 10.4 g of pure *Brachystegia eurycoma* starch was initially suspended in 40 mL of distilled water at 50 ℃ and later instantaneously increased to the investigation temperature by adding 350 mL of hot water at a pre-calculated temperature.

2.3 Viscosity measurement

An NDJ-85 digital display viscometer was used in characterizing the flow behaviour of *Brachystegia eurycoma* flour and starch dispersions. Dispersion samples were first equilibrated to 25 ℃ by dipping them in ice baths with gentle agitation before introducing into the viscometer. Paste viscosity was measured using a spindle No. 2 at 25 ℃, and 10 shear rates: 6, 12, 18, 24, 30, 36, 42, 48, 54, and 60 min^{-1}. The viscosity values of the pastes were obtained by multiplying dial reading by appropriate factors (supplied by the viscometer manufacturer). Experimental data were fitted to Ostwald-de Waele model using Excel 2007 software. The parameters k and n, which are related to consistency and flow index, respectively, were used for comparisons among samples.

2.4 Statistical analysis

Analysis was done in triplicate. Analysis of variance was performed to calculate significant differences in treatment means, and least significant difference (LSD) ($P<0.05$) was used to separate means using SPSS/16 software for windows.

3 Results and discussion

3.1 Flow behaviour of heated *Brachystegia eurycoma* flour and starch dispersions

Good understanding of the rheological behaviours of *Brachystegia eurycoma* starch may not be sufficient to support the use of *Brachystegia eurycoma* as ingredients in the food industry. In most food applications, pure starches of a commodity are rarely used, rather, flour is the common component added as an ingredient during processing. In food processing such as baking, soup making and extrusion, flour materials are the main ingredients; occasionally pure starches are added to enhance texture or nutritional quality. Thus, understanding the rheological behaviour of the flour components is essential, particularly how their individual starch behaviour affects the overall rheological characteristics of the flour during processing.

Rheological changes in heated starch dispersion under isothermal and non-isothermal heating conditions have been widely studied by many researchers (Lund, 1984; Okechukwu et al., 1991; Okechukwu and Rao, 1996; Da Silva et al., 1997; Rao et al., 1997; Thebaudin et al., 1998; Tattiyakul and Rao, 2000; Paterson et al., 2001; Tattiyakul et al., 2002). The relationship of shear stress to shear rate data for isothermally heated *Brachystegia eurycoma* flour (5.0%) and *Brachystegia eurycoma* starch (2.6%), respectively, increased with increasing rate of shear (γ) and temperature. The *Brachystegia eurycoma* flour had higher shear stress, σ, than the *Brachystegia eurycoma* starch at all the rate of shear studied. The increase in shear stress with shear rate is supported by the fact that, regardless of heating temperature and time the apparent viscosity for *Brachystegia eurycoma* flour and starch dispersions decreased with increasing shear rate or increasing shear stress (Figures 1 and 2), exhibited non-Newtonian and shear-thinning behaviour and is produced when the stress disorganized the arrangement of the macromolecules inside of the matrix.

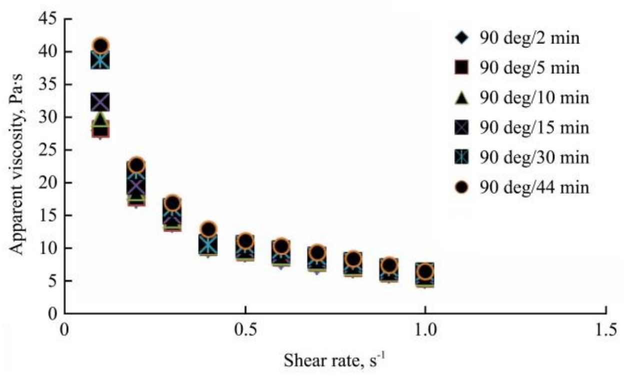


Figure 1 Apparent viscosity vs shear rates curves of *Brachystegia eurycoma* flour dispersion (5.0%) heated at 90 ℃ for 2, 5, 10, 15, 30 and 44 min

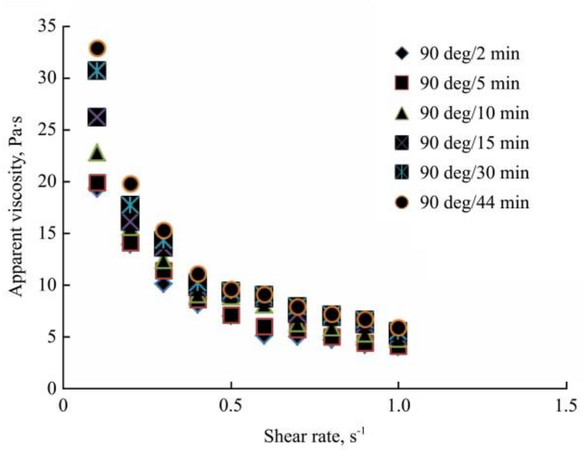


Figure 2 Apparent viscosity vs shear rates curves of *Brachystegia eurycoma* starch dispersion (2.6%) heated at 90 ℃ for 2, 5, 10, 15, 30 and 44 min

Most researchers have reported shear thinning behaviour for maize starch suspension (Evans and Haisman, 1979). The flow behaviour of *Brachystegia eurycoma* flour and starch dispersions was examined using Ostwald-de Waele model ($\sigma = k.\gamma^n$) with high correlation coefficient ($R^2 = 0.9$). k values related to the samples consistency coefficient, and n values related to flow index, were calculated. The *Brachystegia eurycoma* flour and starch dispersions had a flow behaviour index (n) less than 1 (Tables 1 and 2 respectively), although the *Brachystegia eurycoma* flour showed a lower value and more dependent of shear rate than the *Brachystegia eurycoma* starch. In the case of consistency index (k), *Brachystegia eurycoma* flour was higher than the starch. The consistency index and flow behaviour index varied independently with temperature and time of heating. Generally as heating time increased, k, increased while n decreased. This finding is consistent with reported literature that gelatinized starch dispersion heated to about 90 ℃ show shear thinning (n less than 1.0) (Evans and Haisman, 1979; Doublier, 1981; Ellis et al., 1989; Okechukwu and Rao, 1996; Paterson et al., 2001).

Table 1 Power law flow model parameters for *Brachystegia eurycoma* flour dispersion

Sample	Heating time, min	Consistency index, k, Pa s	Flow index, n (Dimensionless)	R^2
75 ℃	2	0.591	0.311	0.965
	5	0.609	0.319	0.944
	10	0.623	0.317	0.938
	15	0.649	0.310	0.948
	30	0.659	0.256	0.952
	44	0.669	0.243	0.931
80 ℃	2	0.618	0.288	0.932
	5	0.631	0.281	0.914
	10	0.648	0.266	0.900
	15	0.671	0.274	0.952
	30	0.683	0.224	0.979
	44	0.715	0.242	0.994
85 ℃	2	0.715	0.327	0.991
	5	0.718	0.329	0.987
	10	0.729	0.312	0.988
	15	0.743	0.302	0.998
	30	0.757	0.249	0.993
	44	0.768	0.234	0.933
90 ℃	2	0.729	0.275	0.992
	5	0.735	0.276	0.988
	10	0.746	0.263	0.995
	15	0.766	0.255	0.999
	30	0.786	0.204	0.995
	44	0.809	0.203	0.995

Table 2 Power law flow model parameters for *Brachystegia eurycoma* starch dispersion

Sample	Heating time, min	Consistency index, *k*, Pa s	Flow index, *n* (Dimensionless)	R^2
75 ℃	2	0.416	0.398	0.980
	5	0.440	0.403	0.962
	10	0.459	0.410	0.952
	15	0.483	0.404	0.950
	30	0.489	0.376	0.963
	44	0.496	0.293	0.975
80 ℃	2	0.538	0.429	0.965
	5	0.548	0.424	0.968
	10	0.563	0.388	0.956
	15	0.578	0.386	0.942
	30	0.580	0.293	0.934
	44	0.596	0.261	0.959
85 ℃	2	0.589	0.384	0.947
	5	0.608	0.391	0.956
	10	0.626	0.333	0.950
	15	0.636	0.320	0.930
	30	0.650	0.278	0.962
	44	0.685	0.260	0.998
90 ℃	2	0.622	0.306	0.947
	5	0.636	0.310	0.965
	10	0.716	0.336	0.959
	15	0.727	0.303	0.975
	30	0.748	0.267	0.994
	44	0.767	0.249	0.997

Because both the consistency coefficient and the flow behaviour index varied independently with time of heating, they were considered unsuitable as variables for determining kinetic parameters (Okechukwu et al., 1991). Therefore, apparent viscosity was examined as an alternative. The patterns of viscosity changes with shear rate for all temperatures investigated for *Brachystegia eurycoma* starch and flour dispersions were examined and, on the basis of greater resolution and sensitivity, apparent viscosities evaluated at the shear rate of 0.1 s^{-1} were chosen for analysis of kinetic data.

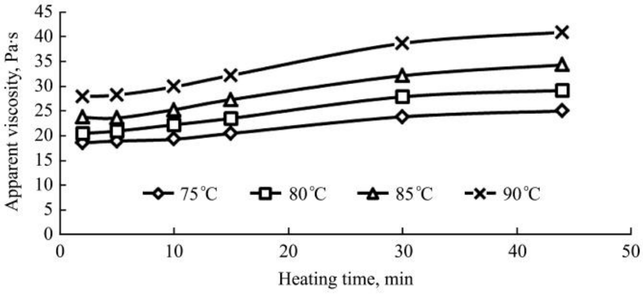
Figure 3 shows apparent viscosity changes with time at various heating temperatures for 5% *Brachystegia eurycoma* flour and 2.6% *Brachystegia eurycoma* starch dispersions, respectively.

The dispersion viscosity was observed to increase with heating time, but did not tend to maximum or limiting value because of the short period of heating time chosen.

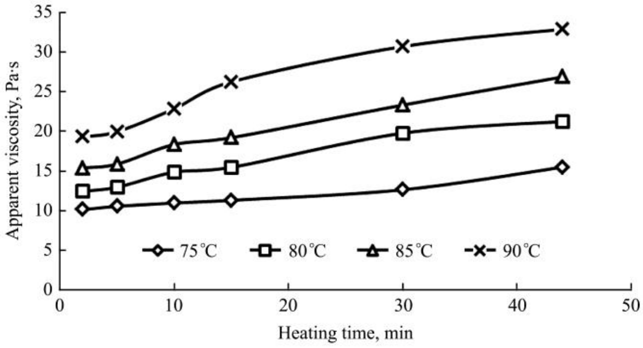
For evaluating the kinetic parameters of gelatinization, the maximum viscosities attained before thinning were regarded as equilibrium viscosities (Okechukwu et al., 1991). The maximum viscosities (μ_∞) were evaluated from the slope of the straight line achieved from the plot of $t\mu_{(t)}^{-1}$ vs t (Equation (1))

$$t\mu_{(t)}^{-1} = k_1\mu_\infty^{-1} + t\mu_{(t)}^{-1} \qquad (1)$$

These limiting equilibrium viscosities were observed to be temperature dependent with higher values at higher temperatures. The attainment of equilibrium viscosities which are temperature dependent is indicative of the heterogeneity of starch granules (Lund, 1984). Similar trends were also reported by Kubota et al. (1979) for potato and rice starches and Okechukwu et al. (1991) for cowpea starch.



a. *Brachystegia eurycoma* flour



b. *Brachystegia eurycoma* starch

Figure 3 Apparent viscosity at 0.1 s^{-1} vs heating time for *Brachystegia eurycoma* flour and starch dispersions heated at 75 ℃, 80 ℃, 85 ℃ and 90 ℃

3.2 Gelatinization kinetics of *Brachystegia eurycoma* flour and starch dispersions

In the cooking of intact materials such as corn and rice grains, moisture diffusion has been identified as a limiting factor in the course of starch gelatinization at high temperatures (Kubota et al., 1979; Suzuki et al., 1976; Cabrera et al., 1984). The use of models for prediction of the viscosity of suspensions of solids have provided useful insights on the flow behaviour of gelatinised starch suspensions as well as of other plant food suspensions (Bagley and Christianson, 1982; Ellis et al., 1989; Okechukwu and Rao, 1995). Gelatinised starch suspension can be regarded as a composite material that is composed of swollen granules and granular fragments that are dispersed in a continuous biopolymer matrix (Evans and Haisman, 1979; Eliasson, 1986; Morris, 1990).

With good temperature control, kinetic evaluation of gelatinization should provide insights into rate processes as well as their controlling factors through such information as reaction order, rate constants, temperature dependence of rate constants and activation energies. It is accepted in literature that the thermal gelatinization of a water/starch system follows first order kinetics (Bakshi and Singh, 1980; Kokini et al., 1992; Okechukwu and Rao, 1996; Okechukwu et al., 1991; Zanoni et al., 1995) by Equation (2).

$$(1-\alpha) = \exp(-Kt) \tag{2}$$

where, α is degree of gelatinization; t is gelatinization time (min) and K is gelatinization rate constant (s^{-1}).

The degree of gelatinization, α, with increasing time is defined as Equation (3);

$$\alpha(t) = [\mu_{(t)}\ \mu_{\infty}^{-1}] \tag{3}$$

where, $\mu_{(t)}$ is viscosity for a partially treated (i.e., gelatinized) sample and μ_{∞} is the maximum viscosity associated with the gelatinization process.

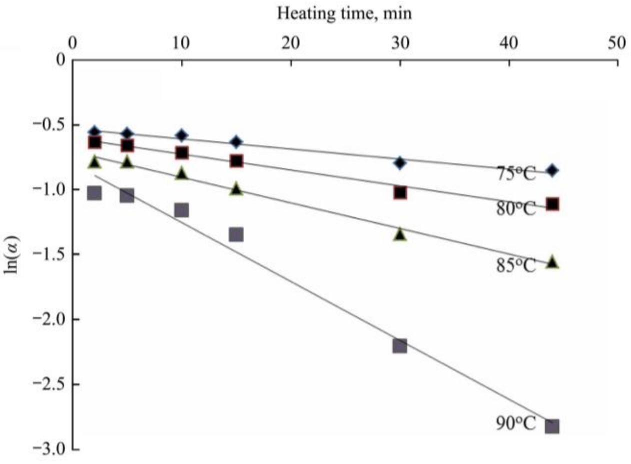
Good linear relationships obtained from the gelatinization rate constant, K, versus heating time, obtained as gradients of the semi log plots for the 5% *Brachystegia eurycoma* flour dispersion ($R^2 = 0.976 - 0.989$) and 2.6% *Brachystegia eurycoma* starch dispersion ($R^2 = 0.945 - 0.997$) are depicted in Figure 4.

The gelatinization rate constant, K, were temperature dependent and increase with increase in temperature within the range 75 ℃-90 ℃. The values of gelatinization rate constants (Table 3) used in Equation 4, were obtained from the intercept (K_0) and the slope (EaR^{-1}) of the linear plots of ln(K) vs T^{-1} (Figure 5) (Equation (5)).

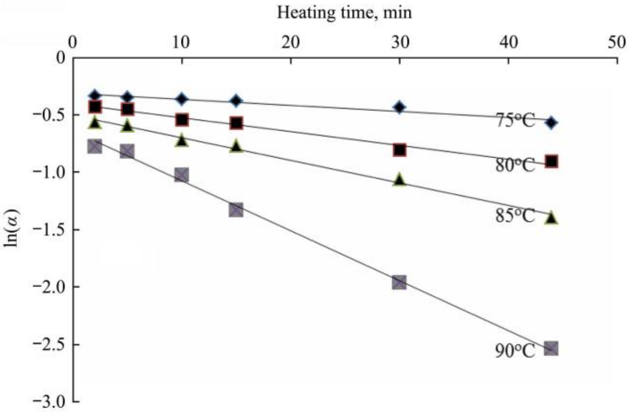
$$K = K_0 \exp[-E_a R^{-1} T^{-1}] \tag{4}$$

where, K_0 is reaction frequency factor; E_a is activation energy of gelatinization (J mol^{-1}); R is gas constant (8.314 J mol^{-1} K^{-1}) and T is absolute temperature (K).

$$\ln(K) = \ln(K_0) + E_a R^{-1} T^{-1} \tag{5}$$



a. *Brachystegia eurycoma* flour



b. *Brachystegia eurycoma* starch

Figure 4 First order kinetics of 5.0% *Brachystegia eurycoma* flour, 2.6% *Brachystegia eurycoma* starch gelatinization

Table 3 Estimates of gelatinization rate constant for 5% *Brachystegia eurycoma* flour and 2.6% *Brachystegia eurycoma* starch dispersions

Temperature, ℃	*Brachystegia eurycoma* flour rate constant, min^{-1}	R^2	*Brachystegia eurycoma* starch rate constant, min^{-1}	R^2
90	0.04528	0.983	0.04330	0.997
85	0.01976	0.989	0.01968	0.993
80	0.01214	0.979	0.01189	0.983
75	0.00773	0.976	0.00522	0.945

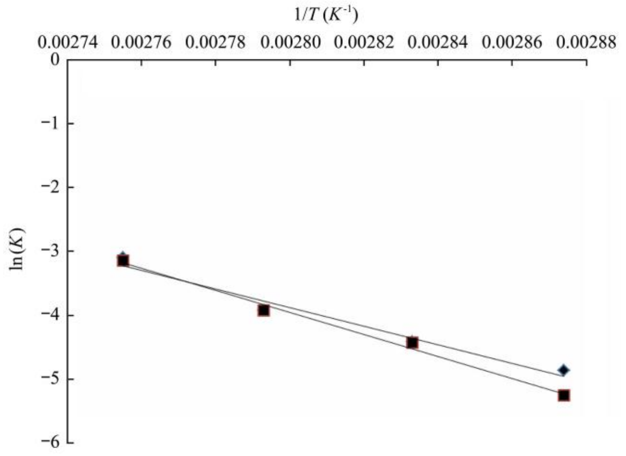


Figure 5 Temperature effect on the gelatinization rate constant of *Brachystegia eurycoma* flour and starch

Figure 5 suggests that the Arrhenius equation described the influence of temperature on the gelatinization rate constant of *Brachystegia eurycoma* flour and starch dispersions reasonably well with the R^2 value, which is a measure of the goodness of fit higher than 0.93. The magnitude of the activation energy (E_a) was estimated to be about 143.43 and 120.91 kJ mol^{-1} for *Brachystegia eurycoma* starch and flour respectively. The activation energy of *Brachystegia eurycoma* starch dispersion was observed to be higher than that of the *Brachystegia eurycoma* flour dispersion. With carbohydrates present in *Brachystegia eurycoma* to the level of 60%-73% (Ikegwu et al., 2010; Enwere, 1998), the high value for activation energy for *Brachystegia eurycoma* starch seemed to suggest the dominant role of starch in the cooking of *Brachystegia eurycoma* dispersions. Since activation energy is a parameter reflecting the sensitivity of the viscosity change due to temperature change, the *Brachystegia eurycoma* starch was more sensitive compared to *Brachystegia eurycoma* flour, hence more heat was needed to break the intermolecular bond as indicated by its higher activation energy. The definition of activation energy implies there is a barrier against viscous flow in *Brachystegia eurycoma* pastes. There has been extensive work carried out on modeling of gelatinization kinetics of grains and isolated grain starches. The activation energy has been reported in the range of 11-960 kJ mol^{-1} depending upon the types of starch and temperature range used for gelatinization (Table 4). Comparison of E_a values for gelatinization of other starches (Table 4) showed that the activation energy of *Brachystegia eurycoma* starch appeared to be higher than that for rice, corn and wheat starches but lower than that for potato and cowpea starches.

Table 4 Activation energy (*Ea*) values for thermal gelatinization of starch obtained by fitting first-order rate constant at various temperatures to the Arrhenius equation

Materials	Temperature, ℃	E_a, kJ mol^{-1}	Reference
White rice	75-110	79.4	Suzuki et al. (1976)
	110-150	36.8	
White rice (Taiwan rice)	48-63.5	62.9	Lin (1993)
	Above 63	11.1	
White rice (Basmati)	60-75	38	Ramesh (2001)
Brown rice (short grain: S6)	50-85	77.4	Bakshi and Singh (1980)
	85-120	43.8	
Rice flour (long grain: Irgo)	<70	287	Ojeda et al. (2000)
	>70	30	
Rice starch	<85	187	Birch and Priestly (1973)
	>85	99	
Rice starch	73-92	42	Kubota et al. (1979)
Potato starch	60-63	961.4	
Potato	<67.5	819.3	Pravisani et al. (1985)
	>67.5	243.3	
Rice starch	<76	306	Yeh and Li (1996)
	>76	43	
Waxy rice starch	59-72	385	Lai and Lii (1999)
Rough rice	30-60	289.3	Bello et al. (2007)
	60-90	16.6	
Hard wheat starch (in situ)	60-75	133	Turhanand Gunasekaran(2002)
	75-100	76	
Corn starch (in situ)	<80	111	Cabrera et al. (1984)
	>80	45	
Cowpea starch	73-85	198.2	Okechukwu et al. (1991)
Brachystegiaeurycoma flour	75-90	120.91	this study
Brachystegiaeurycoma Starch	75-90	143.43	this study

4 Conclusions

Brachystegia eurycoma flour (5%) and starch (2.6%) dispersions heated for 2, 5, 10, 15, 30 and 44 min at 75 ℃, 80 ℃, 85 ℃ and 90 ℃ were characterized by pseudoplastic behaviour ($0.203<n<0.329$) and ($0.249<n<0.429$) respectively with shear thinning effect, and increasing shear stress with shear rate. The consistency coefficient, k, increased with increasing heating time, while the flow behaviour index, n, decreased with increasing heating time. Maximum viscosities attained due to heat-induced gelatinization showed a power relationship with heating temperature. The viscometric measurements showed that starch gelatinization followed a first order kinetic model with the rate constants showing an Arrhenius temperature dependence with an activation energy of 143.43 and 120.91 kJ mol^{-1} for *Brachystegia eurycoma* starch and *Brachystegia eurycoma* flour. The kinetic parameters presented here may be beneficial in food processing when several factors such as inactivation of enzymes and micro-organisms, and retention of colour, aroma components or texture have to be taken into account simultaneously and optimized.

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