

# Studies on some Physicochemical and Rheological Properties of the Plant Gum Exudates of *Albizia furriguinea*

Paul O. Ameh, Umar M. Sani, Elias E. Nwoye

Department of Chemistry, Nigeria Police Academy Wudil, Kano State Nigeria

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## Abstract

Gum exudates from *Albizia furriguinea* have been analyzed for their physicochemical parameters and found to be ionic, mildly acidic, odourless, and yellow in colour. The gum is soluble in water, sparingly soluble in ethanol, and insoluble in acetone and chloroform. The nitrogen (0.53%) and protein (3.498%) contents of the gum are relatively low. The concentrations of the cations were found to increase according to the following trend, Mg > Ca > Mn > Fe > Zn > Pb > Cu > Cd. The scanning electron micrograph of the gum revealed that the gum is irregularly shaped and amorphous. Rheological modeling revealed that the viscosities of the gum vary with temperature, pH and concentration (the viscosity of the gum increases with increasing pH and concentration, but decreases with increase in temperature). The apparent activation energy of flow for the gum calculated from Arrhenius-Frenkel-Eyring plot was found to be 4.16 J/mol suggesting the presence of limited inter and intra molecular interaction. Values of intrinsic viscosity obtained from Huggins and Kraemer plots were similar (11.08 and 8.340 dL/g, respectively) while those obtained from Tanglertpaibul and Roa plots ranged from 7.25 to 21.30 dL/g. The study also revealed that the gum has some potential for use as food additives and for other industrial applications.

**Keywords:** *Albizia furriguinea*, rheology, GCMS, viscosity, physicochemical characterization

## 1. Introduction

Plant gums are obtained as an exudation from fruit, trunk or branches of the trees or after mechanical injury of the plant by incision of the bark, after the removal of the branch, or after invasion by bacteria or fungi [1]. The exudates become hard nodules or ribbons on dehydration to form a protective sheath against microorganisms. These plant products have been collected since about 3000 BC, during the Egyptian civilization from Acacia and gum Arabic trees, native to North - Africa and used as adhesive in hieroglyphic paints and in the embalming of Egyptian mummies. Nowadays, gums have been found to have application in the food industry where emulsifying and stabilizing properties are utilized. Gums are also used in the pharmaceutical and medical fields, in addition to other industries (cosmetic, mining, adhesive paints and inks) [2-3].

The complex and heterogeneous nature of plant gums in terms of their chemical composition makes it very difficult to predict their properties. Yet, their industrial application has to be based on well characterized gum samples, i.e. samples whose quality and safety of application can be assured because their physicochemical properties are well-known. The physicochemical properties of a compound are the measurable physical and chemical characteristics by which the compou-

\* Corresponding author:

nocaseoche@yahoo.com

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and may interact with other systems, and these characteristics collectively determine the quality, applicability or end-use of the compound [4].

The physicochemical properties of some gums have been examined by some researchers. For instance, the physicochemical properties, cationic composition and rheological behavior of exudates from *Ficus glumosa* were investigated by Ameh [5]. The gum was found to be mildly acidic, ionic, soluble in water but insoluble in organic solvents. The trend for the decreasing concentration of elements determined in the gum sample is  $Mg > Ca > Mn > Zn > Fe > Ni > Cu > Cd > Pb$ . Rheological properties of the gums have been adequately modeled using Huggins, Kraemer, Arrhenius, Tanglerpaibul and Rao theories. The results also indicated that the viscosity of the gum increases with increasing pH and concentration but decreases with increase in temperature. Eddy *et al.*, [6] studied gum exudates from *Anogeissus leiocarpus* gum. They discovered that the gum has significant concentration of essential elements (Mg, Ca, Fe, Cu, Mn) and low concentrations of heavy metals (Pb and Cd). The protein content of the gum was also found to be low (5%). Scanning electron micrograph (SEM) of the gum revealed that the gum is irregularly shaped and amorphous. Rheological study indicated that the viscosity of the gum increases with increasing pH and concentration but decreases with increase in temperature. From the plot of speed of rotation *versus* viscosity, viscosity *versus* shear rate and shear stress *versus* shear rate, a non-Newtonian behaviour (dilatant) with characteristic shear thickening property was verified for *Anogeissus leiocarpus* gum. Mhinzi [3] analyzed gum samples from three selected *Albizia* species from Tanzania and determined their commercial potential by comparing their properties with those of *Albizia zygia* and *Acacia* gums. The properties of the gum exudates from *Albizia amara*, *Albizia pertesiana* and *Albizia harveyi* were found to be similar to those of *Albizia zygia* gum except that their aqueous solutions possess slightly lower viscosity and higher levels of tannin. The *Albizia* gums were much less soluble in water than *Acacia* gums; however, their methoxyl contents and acid equivalent weights (AEW) were similar to those of some *Acacia* gums. The physicochemical properties of gum exudates from three *Acacia* tree species (*A. senegal*, *A. sieberiana* and *A. nilotica*) in Batagarawa, Katsina State, were determined and compared by Yusuf [4]. Analysis of all the samples showed no tannin content. Determined cationic composition of the gum samples showed Ca, Mg, Fe, Na and K as the predominant minerals. Cu, Ni, Co, Mn, Cr, Zn and Pb were not detected by this study. He discovered that despite the physicochemical differences among the samples studied, values of physicochemical parameters obtained compared well with those reported in previous studies on *Acacia* gums in many parts of the world.

*Albizia furruginea* (*A. furruginea*) is a species of plant in the Fabaceae family which is commonly called 'Iatandza' in English, 'Doorawamahalbi' in Hausa and 'ayinre-ogo' in Yoruba. *A. furruginea* is a tree, 6-40 m high with a beautiful spreading crown. The main stem of the tree is nearly straight with rough bark which is thick and peels off usually in older trees. The young branches are densely rusty, pubescent or sometimes subtomentose. *A. furruginea* gum is obtained from the incised trunk of the tree. *Albizia spp.* is also capable of producing exudates that are similar to those produced by *Acacia spp.* because it belongs to the same subfamily [7]. These gums were used as coating materials in compression-coated tablets, which degraded, by the colonic microflora, thereby releasing the drug.

This research work is therefore aimed at contributing to the study of gum exudates from other genera, different from *Acacia*, as substitute of gum arabic in its multiple industrial applications by investigating the cationic, surface morphology, physicochemical and rheological properties of *A. furruginea* gum.

## 2. MATERIALS AND METHODS

### 2.1. Collection of Samples

Crude *A. furruginea* gum was obtained as dried exudates from their parent trees grown at Kanya Babba village in Bura Local Government Area of Jigawa State. The plant material had earlier been identified and authenticated in the herbarium Department of Biological Sciences of Ahmadu Bello University, Zaria. The gum was collected from the plant species by tapping during the mid of July and in the day time [8].

### 2.2. Purification of the Gum

The crude sample of *A. furruginea* gum consists of mixture of large and small modules admixed with bark and organic debris. These were hand sorted to remove fragments of bark and other visible impurities and then the gum particles were spread out in the sun to dry for two weeks.

The crude gum was dissolved in cold distilled water, the solution was strained through muslin and then centrifuged, depositing a quantity of dense gel. The straw coloured supernatant liquor was separated and acidified to pH of 2 with dilute hydrochloric acid. Ethanol was then added until it was 80 percent. The gum that precipitated out was removed by centrifugation at 2000 r/min, washed with alcohol followed by ether and was finally dried in a desiccator. The dried flakes were pulverized using a blender and stored in an air tight container.

### 2.3. Physicochemical Analysis

In order to characterize the gum, it was subjected to the following physicochemical tests.

#### 2.3.1. Percentage Yield of the Purified Gum

The dried, precipitated and purified gum obtained from the crude dried exudate was weighed and the percentage yield was expressed in percentage using the weight of the crude gum, as the denominator.

#### 2.3.2. Solubility

The solubility of the gum was determined in cold and hot distilled water, acetone, chloroform and ethanol adopting the method by Eddy *et al* [6]. 1.0 g sample of the gum was added to 50 mL of each of the above mentioned solvents and left overnight. 25 mL of the clear supernatants were taken in small pre-weighted evaporating dishes and heated to dryness over a digital thermostatic water bath. The weights of the residue with reference to the volume of the solutions were determined using a digital top loading balance (ModelXP-3000) and expressed as the percentage solubility of the gum in the solvents [9].

### 2.4. Concentration of Metals

The concentrations of Zn, Mg, Ca, Mn, Fe, Cu, Cd and Pb were determined using Perkin Elmer atomic absorption spectrophotometer (AAS). Calibration curve for each metal was prepared and the concentration of the metals in the analyte was estimated by extrapolation.

### 2.5. Nitrogen and Protein Content

The protein content of the gum was determined using the Kjeldahl method with the nitrogen content being multiplied by a factor of 6.25 [10].

### 2.6. pH

A 1.0 g of the gum sample was dissolved in 100 mL of hot distilled water. The mixture was allowed to stand for 5 min at room temperature before the pH and temperature was recorded using a pre-calibrated pH meter (Oakton pH meter, Model 1100).

### 2.7. Total Soluble Fibre

The total soluble fibre was obtained following the method as described by Sabah El-Kheir *et al.* [11].

### 2.8. Tannin Content

0.1 mL of aqueous FeCl<sub>3</sub> solution was added to 20 mL of a 2 % aqueous solution of the gum sample and the mixture centrifuged. The absence of black precipitate or blackish colouration indicated the absence of tannin [12].

### 2.9. Ash Content

5.0 g of gum sample was heated on a burner in air to remove its smoke. It was further burned in a furnace at 550°C. The ash content was expressed as a % ratio of the mass of the ash to the oven dry mass [13].

### 2.10. Melting Temperature

The melting temperature range of each gum sample was determined using a Gallenkamp melting point apparatus. A 1.0 g of the ground gum was taken in a glass capillary tube and the melting temperature determined repeatedly until reproducible.

### 2.11. Moisture Sorption Studies

The method described by Josiah [14] was adopted. Dried evaporating dishes were weighed and 2.0 g of each of gum samples was weighed into the different dishes. The final weight of the dishes was noted and placed over water in desiccators for a period of 5 days. They were thereafter removed and transferred into other desiccators over activated silica gel (desiccant) for another 5 days. The dishes with their various contents were weighed on daily basis and their water content calculated.

### 2.12. Swelling property

In order to study the swelling property of the gum, the method reported by Ohwoavworhua and Adelokun [15] was

adopted. The sample (1.0 g) was placed in a 15 mL plastic centrifuge tube and the volume occupied was noted. Distilled water (10 mL) was added from a 100 mL measuring cylinder and stoppered. The contents were shaken thoroughly for 2 min and further allowed to stand for 10 min. Each sample was centrifuged at 1000 rpm for 10 min on a bench centrifuge. The supernatant was then decanted and the volume of sediment obtained was measured. The swelling property of the gum was calculated using Equation 1.

$$S = V_2/V_1 \quad 1$$

where  $S$  is the swelling index,  $V_1$  is the volume occupied by the gum prior to hydration and  $V_2$  is the volume occupied by the gum after hydration.

### 2.13. Viscosity Measurements

The apparent viscosity of the mucilage was measured using a digital Brookfield DV I prime viscometer. The intrinsic viscosity of the gum samples was determined in distilled water. The gum solutions were prepared by dispersing 50 mg of each gum sample (db, dry basis) separately in 100 mL of the distilled water at room temperature and mixed using a magnetic stirrer overnight. The solution (2 mL) was transferred into a Cannon Ubbelohde capillary viscometer (Cannon Instruments, Model I-71) which was immersed in a precision water bath to maintain the temperature at  $25.0 \pm 0.1^\circ\text{C}$  and after equilibration for 10 min, the flow time was determined between the two etched marks. Serial dilution was performed in situ and three readings were taken for each dilution and the average obtained. The relative viscosity ( $\eta_{\text{rel}}$ ) was calculated using Equation 2.

$$\eta_{\text{rel}} = (T - T_o)/T_o \quad 2$$

where  $T$  is the flow time of gum solution in seconds,  $T_o$  is the flow time of solvent (water) in seconds.

Microsoft Excel 2010 (Microsoft Corporation, Seattle, WA) was used to plot viscosities against concentrations, as well as to obtain linear regression lines with the corresponding equations and correlation coefficients ( $R^2$ ) in order to assess the best model.

### 2.14. Scanning Electron Microscopy

The morphological features of the gum were studied with a JSM-5600 LV scanning electron microscope. The dried sample was mounted on a metal stub and sputtered with gold in order to make the sample conductive and the images were taken at an accelerating voltage of 10 kV.

## 3. RESULTS AND DISCUSSIONS

### 3.1. Physicochemical Properties of the Gum

Table 1 presents the physicochemical parameters of *A. furrigunea* gum. The physical parameters examined included colour, odour, pH, solubility in water and taste. From the results obtained, it is evident that the colour is yellowish brown to pure yellow (Figure 1). The gum was also found to be odourless. From the measured pH values of the gum samples, it was found that the acidity of *A. furrigunea* gum is mild, indicating weak acidity. The acidity of the plant gum is not unexpected; it is known to contain salts (K, Na, Ca, Mg, and Fe) of acidic polysaccharides, the acidity of which is due to uronic acids in their structures [1, 2, 6, 16].



**Figure 1.** Photographs of Crude (a) and Purified (b) Samples of *A. furrigunea* Gum.

The nitrogen content of *A. furrigunea* gum was found to be 0.53%. Since the protein content of the gums is directly related to the nitrogen content, it is expected to follow the trend similar to that of nitrogen content, found in the present result. It has also been established that film forming, emulsifying and stabilizing properties of a gum are related to the protein fraction [17]. Therefore, the nitrogen contents of gums can also be used as an index for examining the food value.

The gum sample was found to be soluble in cold and hot water. However, the solubility of the gum in hot water was found to be higher than the corresponding solubility in cold water. The observed increase in solubility with temperature indicate that the heat given off in dissolving the gum is less than the heat required to break the gum apart. The samples were not soluble in acetone and chloroform but were sparingly soluble in ethanol. This indicates that the gum is ionic rather than covalent [18].

No tannin content was found in the gum sample, consistent with results obtained by many researchers who worked on the chemical composition of most gums [17, 19-20].

Ash content of the gum was found to be 3.35% which falls within the 4% maximum limit reported by FAO [12] for food and pharmaceutical quality gums.

**Table 1.** Physicochemical and Rheological Properties of *A. furrigunea* Gum

Parameters	<i>Albizia furrigunea</i>
Colour	Yellow
Odour	Odourless
Taste	Bland
pH	4.8
Solubility % w/v in	
cold water	7.7
Hot water	7.4
Acetone	0.00
Chloroform	0.00
Ethanol	0.00
Intrinsic viscosity ( $\eta$ ), dL/g	11.080
Nitrogen (%)	0.53
Protein (%)	3.498
Percentage yield(% w/w)	75.4
Swelling capacity	9.90
Melting temperature ( $^{\circ}$ C)	298-310
Tannin content	0.00
Total soluble fibre (%)	78.4
Ash (%)	3.35

### 3.2. Water Sorption Capacity

Figure 2 shows the plots for the variation of the water sorption capacity of the gum with time. From the figure, it is apparent that the water absorption capacity increased progressively up to the fifth day of immersion (i.e. 100 % relative humidity over water) and dropped sharply within 24 h when subjected to action of a desiccant. By the fifth day in a desiccant environment, the water content of the gum had reduced considerably to between 1-4%. The results obtained show that if the gum is stored in a damp environment, the gum will quickly be hydrated with a tendency to rapidly loose such water molecules in the presence of desiccants (within five days). The observed results are consistent with the findings by Abdulsamad *et al.* [21] for cashew and *acacia* gums. Generally, susceptibility to microbial and physicochemical deterioration as a result of high moisture content may be some of the factors that can be associated with the water sorption potentials of the studied gum. Therefore, this gum can better be preserved in an air-tight container.

### 3.3. Rheological Properties

The effect of pH on the viscosity of the gum sample was investigated (Figure 3). It can be deduce from the figure that the viscosity of the gum tend to increase with increase in pH indicating that the emulsifying properties of the gum is pH dependent [22].

The increase in viscosities of the gum with increase in pH also indicates that the gum is ionic [23]. Viscosity of a liquid is related to the ease with which the molecules can move with respect to one another. Thus, the viscosity of a liquid depends on the strength of attractive forces between molecules, which in turn depend on their composition, size, shape and also on the kinetic energy of the molecules, which depend on the temperature. This implies that the strength of hydrogen bonding between the molecules of the gum can partly be a factor accounting for the increase in viscosity with pH. At higher pH (that is increasing alkalinity), the viscosity of the gum is expected to increase.

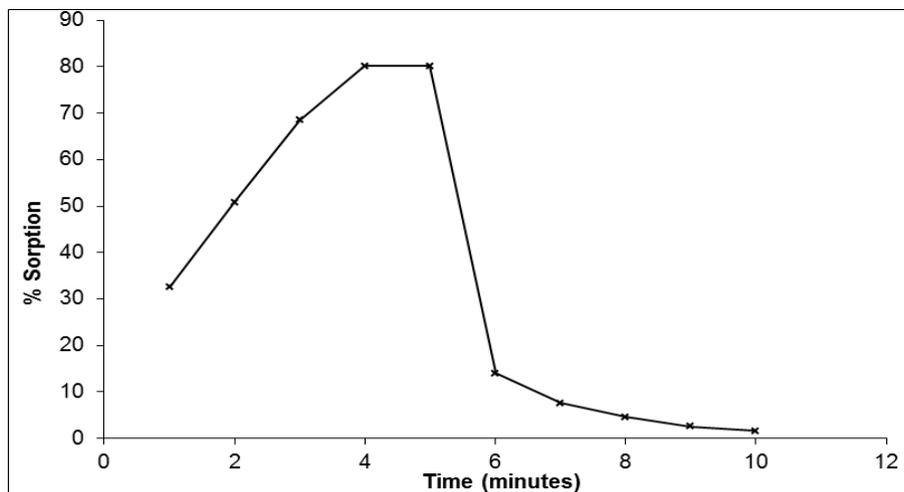


Figure 2. Variation of Water Sorption Capacity with Time for *A. furrigunea*

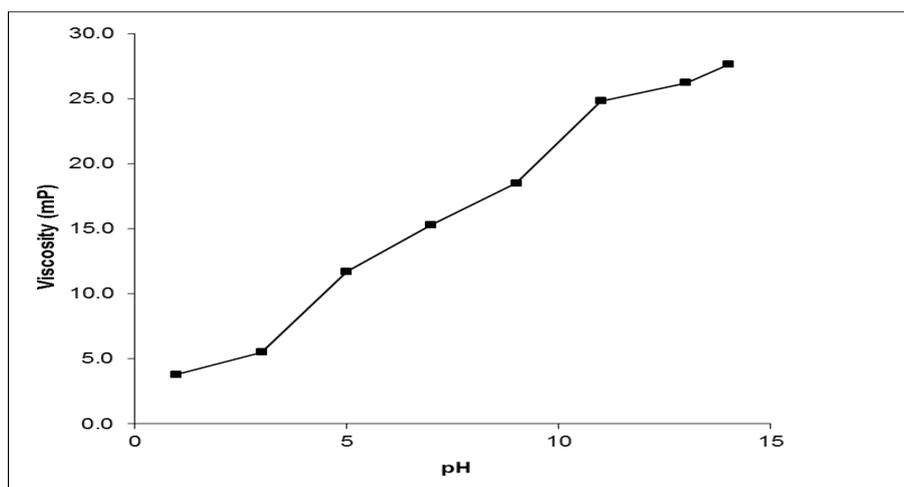


Figure 3. Variation of Viscosity with pH for *A. furrigunea* Gum

In Figure 4, the plot for the variation of viscosity with temperature for the gum is presented. Values of viscosity were recorded between the temperatures ranges of 28 to 40°C. It can be seen from the plot that the viscosity of the gum decreases with increasing temperature. This trend occurs because the increased kinetic motion at higher temperatures promotes the breaking of intermolecular bonds between adjacent layers. The temperature dependence of viscosity is the phenomenon by which viscosity tends to decrease (or alternatively, its fluidity tends to increase) as its temperature increases.

Several models have been proposed to explain the dependence of viscosity on temperature. The exponential model states that the viscosity of a system varies exponentially with temperature as shown in Equation 3.

$$\eta(T) = \eta_0 \exp(-bT) \tag{3}$$

where  $T$  is temperature and  $\eta_0$  and  $b$  are coefficients. The logarithm of both sides of Equation 3 yields Equation 4.

$$\log \eta = \log \eta_0 - bT \tag{4}$$

From Equation 4, a plot of  $\log \eta$  versus  $T$  should be linear provided the exponential model is obeyed. Figure 5 shows the exponential plots for the viscosity of the gum. Values of  $b$ ,  $\eta$  and  $R^2$  deduced from the plots are presented in Table 2. From the results obtained, it is evident that  $R^2$  values are above average confirming the application of the empirical model to the variation of viscosity of the gum with temperature. One of the major shortcomings of the empirical model is that it is applicable to a limited range of temperatures (i.e > 303 K), above which the model is not obeyed [24].

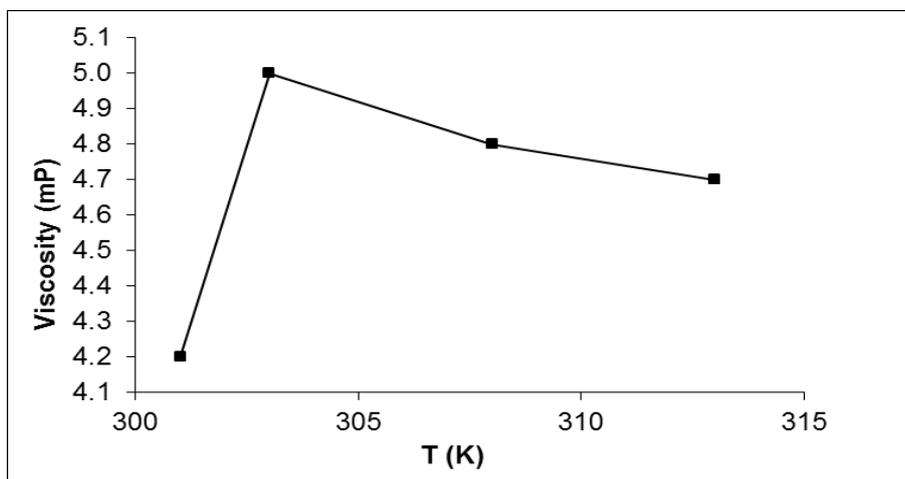


Figure 4. Variation of Viscosity with Temperature for *A. furrigunea* Gum

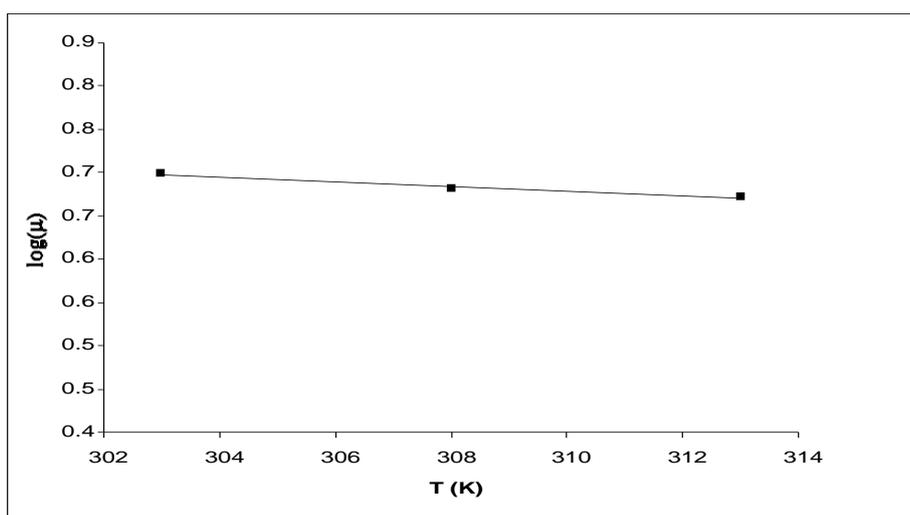


Figure 5. Variation of  $\log(\text{viscosity})$  with Temperature for *A. furrigunea* Gum

Arrhenius model is one that can be used to study the relationship between viscosity and temperature. This model is based on the assumption that fluid flow obeys the Arrhenius equation for molecular kinetics and can be written as Equation 5.

$$\eta(T) = \eta_0 \exp(E_a/RT) \quad 5$$

where  $T$  is temperature,  $\eta_0$  is the Arrhenius coefficient,  $E_a$  is the activation energy and  $R$  is the universal gas constant. A first-order fluid is another name for a power-law fluid with exponential dependence of viscosity on temperature [24]. From the logarithm of Equation 5, a plot of  $\log \eta$  versus  $1/T$  should be a straight line with intercept and slope equal to  $\eta_0$  and  $E_a/R$ , respectively.

Table 2. Empirical Model Parameters for the Viscosity *A. furrigunea* Gum

Gum	b	$\mu_0$	$R^2$
<i>A. furrigunea</i>	-0.0027	1.512	0.9671

Figure 6 shows the Arrhenius plot for molecular kinetics of the gum. Values of the activation energy and  $\eta_0$  deduced from the plots are recorded in Table 3. From the results obtained, it is evident that values of  $b$  deduced from the empirical model plot (Figure 5) approximate values of  $E_a$  obtained from the Arrhenius molecular kinetic model, indicating that the physical meaning of  $b$  is the activation energy of the gum. The low values of  $E_a$  obtained suggests the existence of few inter and intra-interactions between the molecules of the gum within the investigated temperature.

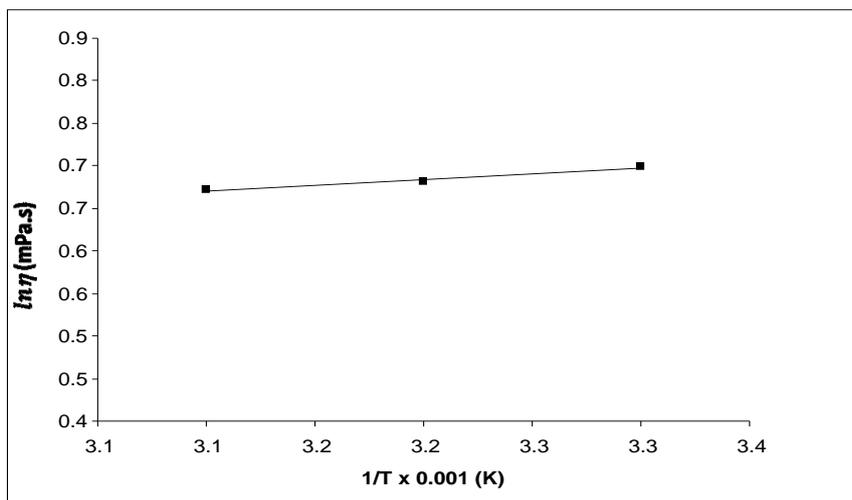


Figure 6. Arrhenius Molecular Kinetic Plots of *A. furrigunea* Gum

Table 3. Arrhenius Parameters for Molecular Kinetic Models of *A. furrigunea* Gum

Gum	$E_a$ (J/mol)	$\eta_0$	$R^2$
<i>A. furrigunea</i>	0.0027	1.512	0.9671

Figure 7 shows the plot for the variation of viscosity with concentration of *A. furrigunea* gum. From the figure, it is evident that the viscosity of *A. furrigunea* gum tends to increase with increase in concentration. All polymers increase the viscosity of the solvent in which they are dissolved [25]. This increase allows for a convenient method of determining the molecular weight of polymers. Since the viscosity method is not based on rigorous physical laws, it must be calibrated by standards of known molecular weights with narrow molecular weight distributions.

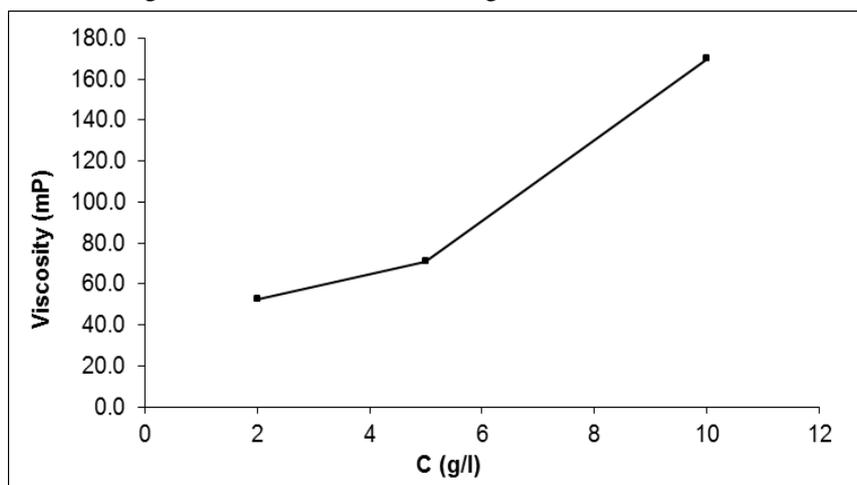


Figure 7. Variation of Viscosity with Concentration of *A. furrigunea* Gum

The intrinsic viscosity,  $[\eta]$  is the limit of the reduced viscosity as the polymer solute concentration approaches zero.

$$[\eta] = \lim_{C \rightarrow 0} \frac{\eta_{sp}}{C} \tag{6}$$

Also, the intrinsic viscosity of a polymer, which can be determined experimentally, is a power series in concentration and can be written as Equation 7.

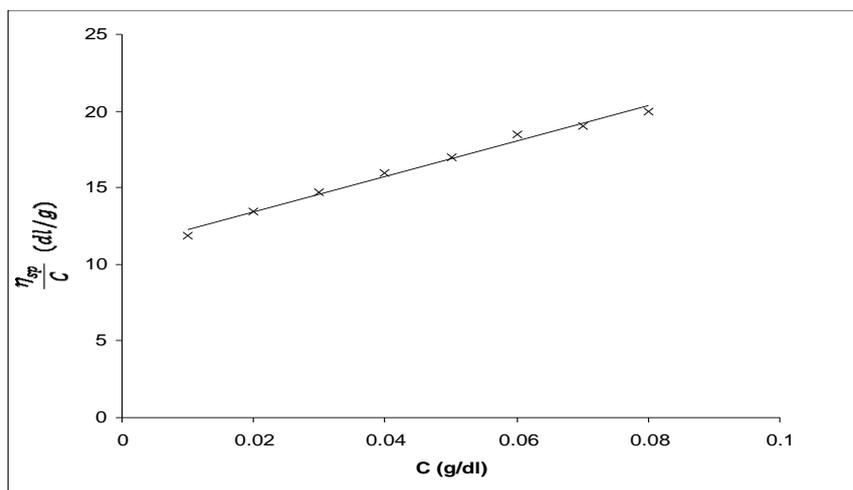
$$\eta_{spec}/C = [\eta] + k_1[\eta]^2C + k_2[\eta]^3C^2 + k_3[\eta]^4C^3 + \dots \tag{7}$$

where  $[\eta]$  is the intrinsic viscosity,  $\eta_{spec}$  is the specific viscosity.  $k_1, k_2, k_3, \dots$  are dimensionless constants. Since  $\eta_{spec}/C$

is a reduced viscosity, which as  $C \rightarrow 0$  becomes the intrinsic viscosity, the above power series is often truncated to a linear approximation known as the Huggins equation [26].

$$\eta_{\text{red}} = [\eta] + k'[\eta]^2 C \quad 8$$

where  $[\eta]$  is the intrinsic viscosity and  $k'$  is the Huggin constant. From Equation 8, a plot of  $\eta_{\text{red}}$  versus  $C$  is expected to be a straight line with intercept equal to  $[\eta]$  and slope equal to  $k'[\eta]^2$ . Figure 8 shows the Huggins plot for the gum. Values of Huggins constant deduced from the plot are presented in Table 4. From the results obtained, values of  $[\eta]$  and  $k'$  approximates 11.083dL/g and 0.945, respectively. This indicates that the degree of polymer-polymer interaction in the studied gum is relatively high.

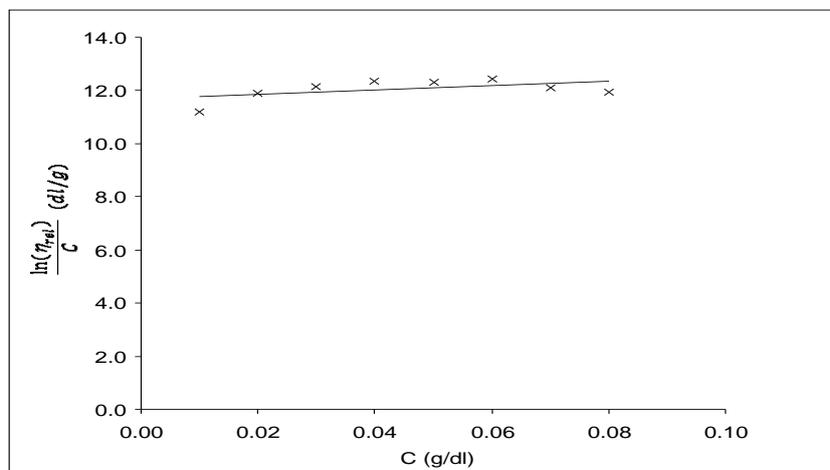


**Figure 8.** Variation of Reduced Viscosity with Concentration (Huggins Plot) for *A. furrigunea* Gum.

The intrinsic viscosity was also analyzed using the Kraemer equation, which is given in Equation 9 [27].

$$\ln(\eta_{\text{rel}}/C) = [\eta] + k''[\eta]^2 C \quad 9$$

where  $k''$  is the Kraemer constant. From Equation 9, a plot of  $\ln(\eta_{\text{rel}}/C)$  versus  $C$  should be a straight line with intercept and slope equal to  $[\eta]$  and  $k''[\eta]^2$ , respectively. Figure 9 shows the Kraemer plot for the gum. Values of the Kraemer parameters deduced from the plot are also recorded in Table 4. From the results obtained,  $k''$  values obtained from Kraemer plot are comparable to  $k'$  values obtained from Huggins plot. However, values of  $[\eta]$  deduced from Huggins plot are relatively higher than those obtained from Kraemer plot.



**Figure 9.** Variation of  $\ln(\text{relative viscosity})$  with Concentration (Kraemer plot) of *A. furrigunea* Gum

**Table 4.** Huggins and Kraemer Parameters for *A. furrigunea* Gum.

	R <sup>2</sup>	Slope	[η]	[η] <sup>2</sup>	k
Huggins	0.9890	11.642	11.083	122.833	0.945
Kraemer	0.9621	8.340	11.675	136.306	0.061

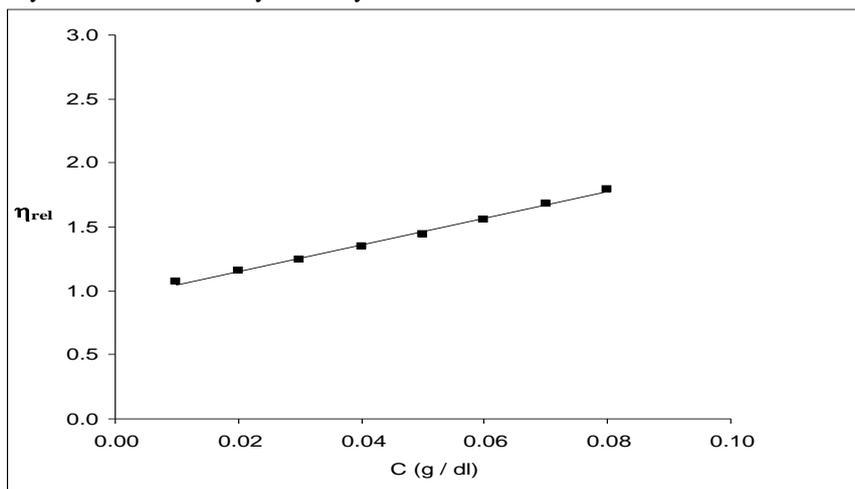
Huggins and Kraemer plots are based on the extrapolation to zero concentration indicating that it is based on the calculation of intercepts in the plots. McMillan [28] showed that methods of determination for the intrinsic viscosity that were based on slopes of plots had higher correlation coefficients and lower standard errors, compared to those based on intercepts of plots. On the bases of such findings, Tanglertpaibul and Rao [29] obtained three equations that can also be used in the determination of the intrinsic viscosity of a polymer. These are Equations 10, 11 and 12.

$$\eta_{rel} = 1 + [\eta]C \tag{10}$$

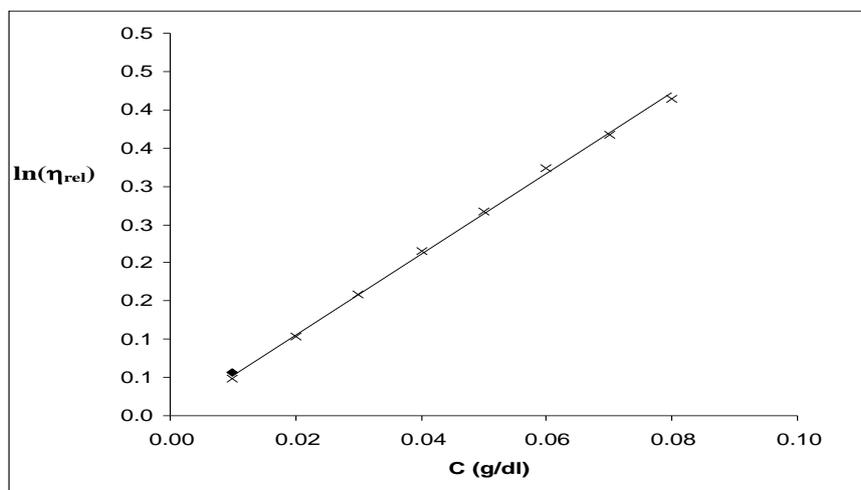
$$\eta_{rel} = \exp([\eta]C) \tag{11}$$

$$\eta_{rel} = 1/(1-[\eta]C) \tag{12}$$

From Equation 10, [η] is the slope obtained by plotting η<sub>rel</sub> versus C. Also, the implication of Equation 11 is that [η] is the slope obtained from the plot of lnη<sub>rel</sub> versus C and from Equation 12, [η] is a slope obtained by plotting (1-1/η<sub>rel</sub>) versus C. Tanglertpaibul and Rao plots for the gum (from Equations 10 to 12) are presented in Figures 10, 11 and 12, respectively. Values of Tanglertpaibul and Rao parameters deduced from the plots are presented in Table 5. Correlation coefficients for the five methods employed in the calculation of intrinsic viscosities of the gum are presented in Table 6. It can be seen that there is an excellent correlation between the values of [η] computed from the various methods indicating the consistency of the methods for the study of intrinsic viscosity of the system.



**Figure 10.** Variation of η<sub>rel</sub> with Concentration of *A. furrigunea* Gum



**Figure 11.** Variation of ln(η<sub>rel</sub>) with Concentration of *A. furrigunea* Gum

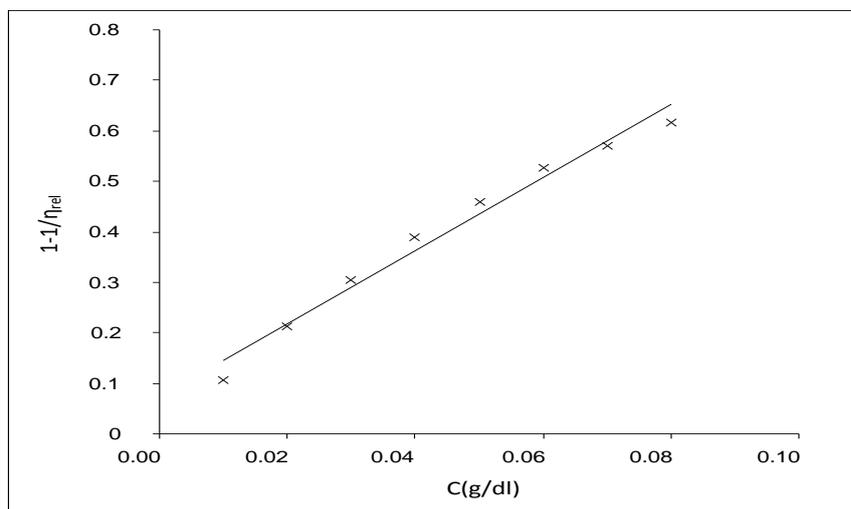


Figure 12. Variation of  $(1 - 1/\eta_{rel})$  with Concentration of *A. furrigunea* Gum

Table 5. Tanglertpaibul and Rao parameters for *A. furrigunea* Gum

Eq. 10		Eq. 11		Eq. 12	
$[\eta]$	$R^2$	$[\eta]$	$R^2$	$[\eta]$	$R^2$
21.308	0.9924	12.159	0.9986	7.2532	0.978

Table 6. Correlation Coefficients for the Various Methods Employed in the Calculation of Intrinsic Viscosities of the Gum

	Huggins	Kraemer	Eq. 10	Eq. 11	Eq. 12
Huggins	1.0000				
Kraemer	0.9981	1.0000			
Eq. 10	0.9629	0.9778	1.0000		
Eq. 11	0.9584	0.9742	0.9993	1.0000	
Eq. 12	0.9444	0.9624	0.9955	0.9982	1.0000

According to Higiroy *et al.* [27], the power law equation relating specific viscosity and concentration can be expressed as Equations 13.

$$\eta_{spec} = aC^b \quad 13$$

The logarithm of both sides of Equation 13 and rearrangement yield Equation 14.

$$\ln(\eta_{spec}) = \log(a) + b \log C \quad 14$$

From Equation 14, a plot of  $\ln(\eta_{spec})$  versus  $\log C$  should give a straight line with slope and intercept equal to 'b' and  $\log(a)$ , respectively. Figure 13 shows the power-law plot for *A. furrigunea* gum. Table 7 presents values of the power law parameters deduced from the plot. From the result obtained, it can be seen that values of  $b$  is 1.257. According to Lar *et al.* [30], the value of  $b$  is an index that can be used to predict the conformation of a polymer. Also, Lapasin and Prici [31] found that  $b$  value greater than unity is associated with random coil conformation while  $b$  value less than unity is associated with rod like conformation. Therefore, the molecular conformation of this gum is more random coil like than rod like.

Table 7. Power Law Parameter for *A. furrigunea* Gum

Gum	B	$\ln(a)$	a	$R^2$
<i>A. furrigunea</i>	1.257	3.620	37.338	0.9992

In order to study the effect of centrifugal forces on the viscosity of the gum, the viscosity of the gum was measured at various speeds of rotation. Figure 14 shows the plot of viscosity versus the rotation speed of a centrifuge. From the Figure 14, it can be seen that the viscosity of the gum tend to decrease with increasing speed of rotation until a critical value is reached after which the viscosity increased with increase in speed of rotation.

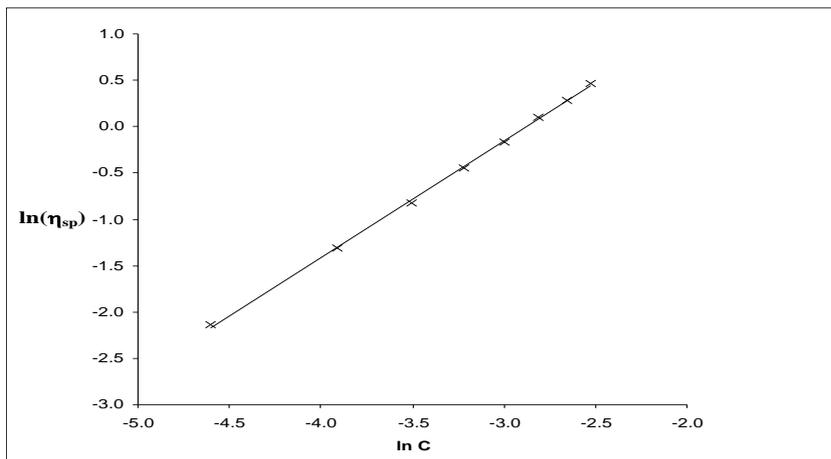


Figure 13. Power Law Plot of *A. furrigunea* Gum.

Several explanations may be ascribed to the observed behaviour of the gum. One of such explanations is the increase in the extent of shaking as a result of increasing speed of rotation. Gundlah and Kapur [32] found that the viscosity behaviour of polystyrene fractions in benzene and methyl ethyl ketone is markedly dependent on the extent of shaking of the solutions and concluded that the anomalous viscosity behaviour of polymers at high dilutions results from configurational changes of the macromolecules.

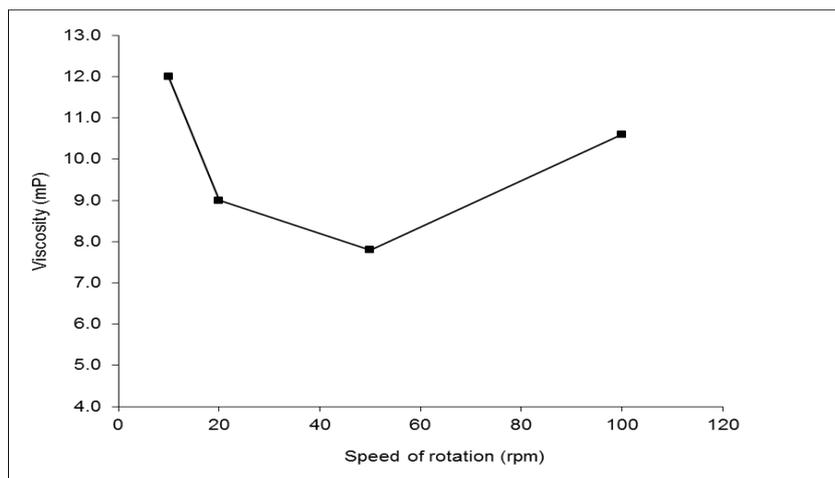


Figure 14. Variation of Viscosity with Rotation Speed of *A. furrigunea* Gum.

In hydrodynamic studies of viscosity, the Taylor number is an index that can be used to characterize the importance of centrifugal forces due to rotation on the viscosity of the system under study. The Taylor number can be defined as follows:

$$T_a = \frac{4\Omega^2 R^4}{\eta^2} \tag{15}$$

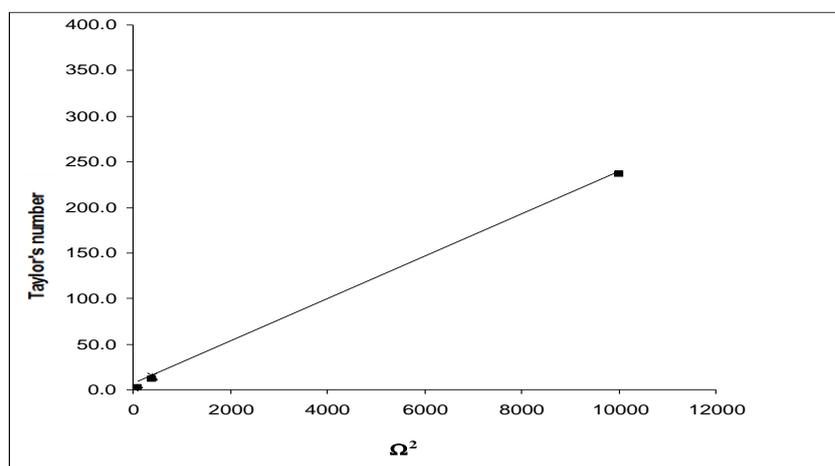
where  $\Omega$  is a characteristic angular velocity,  $R$  is a characteristic linear dimension perpendicular to the rotation axis, and  $\eta$  is the kinematic viscosity. Rearrangement of Equation 15 yields Equation 16

$$\eta^2 = \frac{4\Omega^2 R^4}{T_a} \tag{16}$$

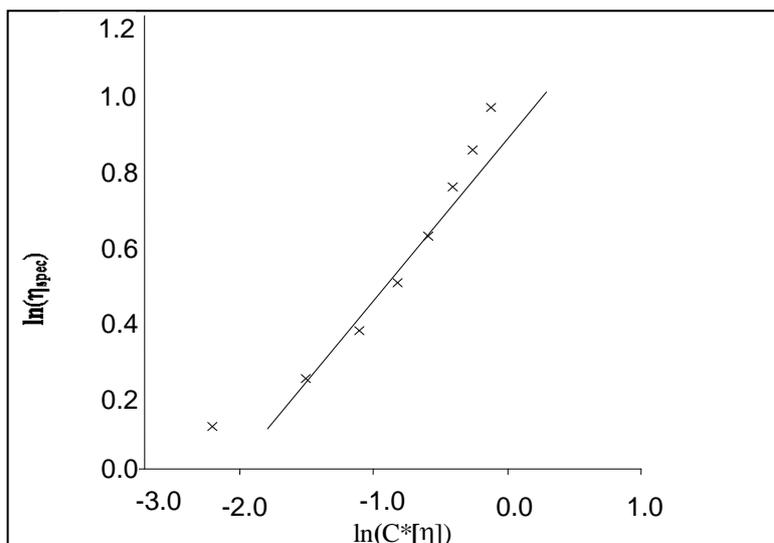
From the above equations, it is evident that the viscosity of macromolecules such as gum is expected to increase as the Taylor number decreases and vice versa. The calculated value for the Taylor number of the gum at various speeds of rotation is presented in Table 8. From the results obtained, it can be seen that the Taylor's number increases with increasing speed of rotation. Also, from the calculated values of  $T_a$ , the plot of  $T_a$  versus  $\Omega^2$  (Figure 15) is linear ( $R^2 = 0.9910$ ) indicating that the viscosity of the gum obtained by varying the speed of rotation is consistent with the Taylor's number.

**Table 8.** Taylor's Number ( $T_a$ ) and Viscosity of the *A. furrigunea* Gum at Various Speed of Rotation

Speed of rotation	$\eta$	$T_a$
10	12.0	2.1
20	9.0	11.1
50	7.8	80.1
100	10.6	235.9

**Figure 15.** Variation of Taylor's Number with Angular Speed of Rotation for *A. furrigunea* Gum.

Based on the variation of shear rate with viscosity, colloidal systems such as gums can be classified as Newtonian or non-Newtonian. A Newtonian fluid is one in which the viscosity is independent of the shear rate. In Newtonian fluids all the energy goes into sliding molecules by each other. In non-Newtonian fluids, the shear stress/strain rate relation is not linear and typically the viscosity drops at high shear rates. In order to characterize the gum as Newtonian or non-Newtonian, the 'master curve' (Figure 16) was developed. The slope of the line on the master plot is 0.9 ( $R^2 = 0.9317$ ). The calculated slope is within the range of values associated with most food gums [5, 8, 33].

**Figure 16.** Variation of  $\log(\eta_{\text{spec}})$  with  $\ln(C^*[\eta])$  of *A. furrigunea* Gum

In order to determine the Newtonian or non-Newtonian characteristics of gums, several methods have been reportedly used. However, one of the most acceptable methods for analyzing non-Newtonian flow involves the generation of a plot of viscosity versus spindle speed using same spindle. If such plot is linear, then the fluid is said to be non-Newtonian. Figure 17 shows the plot of viscosity versus shear rate for *A. furrigunea* gum. The  $R^2$  value (0.9423) obtained from the plot is high indicating that the gum is a non-Newtonian fluid. Also from the plot, the yield stress (i.e. the amount of force needed to be applied to the gum before it can flow) can be estimated. A simple method for determining a relative yield is to plot viscosity readings on the x-axis vs. speed (rpm) on the y-axis. The line obtained, can then be extrapolated to zero rpm. The corre-

sponding value for the viscometer reading represents the yield. Once a straight line is obtained the angle this line forms with the y-axis is measured [34].

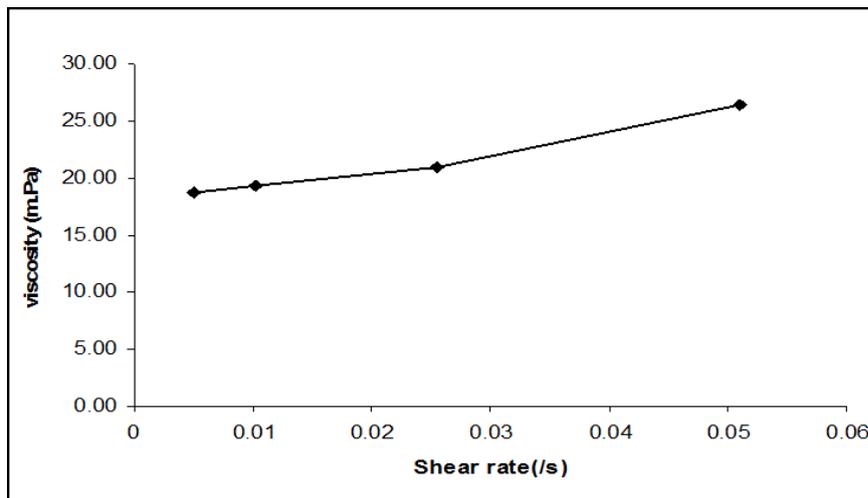


Figure 17. Variation of Viscosity with Shear Rate for *A. furrigunea* Gum

From the measured angle and intercepts, the power law index (N) of this fluid can then be calculated from the equation; Power law index  $N = \tan$  (the angle between the plot line and y-axis) [35].

If the angle is less than 45 degrees, the fluid is pseudoplastic, if greater than 45 degrees, then it is dilatants [35]. The power law index (N) can also be used to calculate the effective shear rate at a given speed using Equation 17 [34].

$$S = N / (0.2095 \times \text{viscometer speed in rpm}) \tag{17}$$

Values of yield stress,  $N$  ( $\theta$ ) and  $\theta$  deduced from the plot in Figure 17 are presented in Table 9. From the results obtained, it can be seen that  $\theta$  values is greater than  $45^\circ$  for the gum. Therefore *A. furrigunea* gum is dilatant.

Table 9. Some Non-Newtonian Parameters for *A. furrigunea* Gum

Gum	Yield stress	$\tan\theta$	$\theta$ ( $^\circ$ )
<i>A. furrigunea</i>	25	8.853	83.56

Figure 18 shows the plot for the variation of shear stress with shear rate for *A. furrigunea* gum. The shape of the graph obtained further confirms that the gum is dilatant and is therefore a shear thickening fluid.

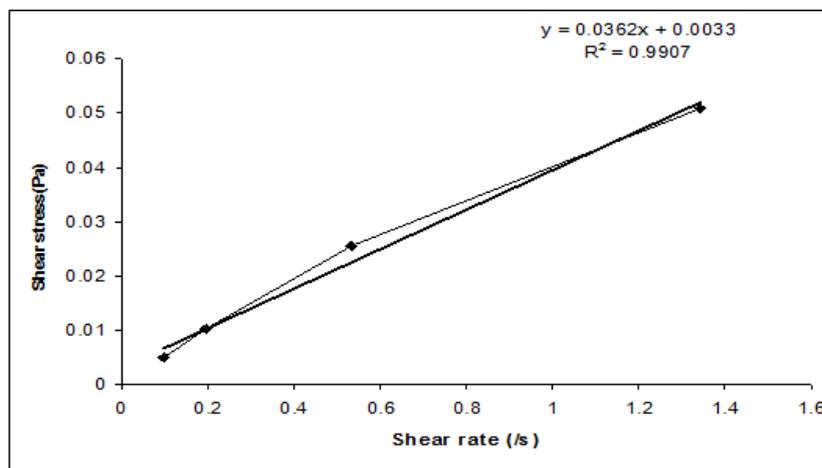


Figure 18. Variation of shear stress with shear rate for *A. furrigunea* Gum

### 3.4. Mark–Houwink equation

The Mark-Houwink equation describes the dependence of the intrinsic viscosity of a polymer on its relative molecular mass (molecular weight) and can be expressed as Equation 18.

$$[\eta] = KM^a \quad 18$$

where  $[\eta]$  is the intrinsic viscosity,  $K$  and  $a$  are Mark-Houwink constants, the values of which depend on the nature of the polymer and solvent as well as on temperature and  $M$  is usually one of the relative molecular mass averages;  $a$  is a function of polymer geometry, and varies from 0.5 to 2.0. However, for the gum, the value of  $a$  used was 0.55 [36].

The calculated value of the molecular weight of the gum obtained from Equation 18 is  $2.78 \times 10^5$ . This value is quiet consistent with others previously reported for other gums [37-40].

### 3.5. Cationic Composition of the Gum

Table 10 presents the concentration of elements identified in the gum sample. From the results obtained, it can be seen that the trend for the decreasing concentration of elements in the gum samples is  $Mg > Ca > Mn > Fe > Zn > Pb > Cu > Cd$ . The high level of Mg shows that the exudate could provide an alternative source of the elements in diets. Mg is required in large quantities by the body for the activation of enzymes involved in protein synthesis. The possible outcomes of deficiency are growth failure. The exudates were also found to contain Mn and Fe. Fe is an essential element needed by the body as its constituent of hemoglobin and enzymes involved in energy metabolism in iron transport [41]. Fe is an element needed by humans in protein synthesis and in the formation of some blood cells.

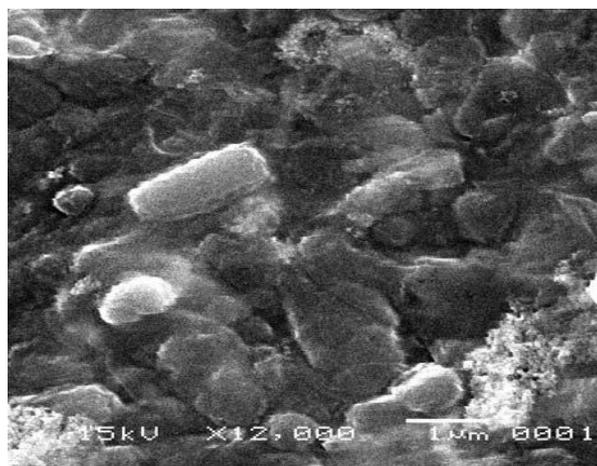
The result also shows that gum is poorer in heavy metal concentrations such as Pb and Cd. Therefore, the gum may be a good source of mineral nutrition and are suitable for use as pharmaceutical excipients and food additives [42].

**Table 10.** Cationic Composition of *A. furriginea* Gum

Element	<i>A. Furriginea</i>
Mg (% w/w)	0.51
Ca (% w/w)	0.97
Zn (ppm)	38.60
Mn (ppm)	120.40
Fe (ppm)	82.70
Cu (ppm)	4.60
Cd (ppm)	1.50
Pb (ppm)	5.30

### 3.6. Scanning Electron Microscopy

Figure 19 shows the SEM of *A. furriginea* gum. The SEM is shown at 1200 x magnification and 1  $\mu$ m scale. It is evident from the figure that the molecules of the gum are tiny granules and slightly elongated with rugged appearance. The micrograph is indicative of an amorphous material and can serve as pharmaceutical excipients [43].



**Figure 19.** Scanning Electron Micrograph of *A. furriginea* Gum

## 4. CONCLUSIONS

- *A. furrigunea* gum is ionic, mildly acidic, odourless, and yellow in colour. The gum is soluble in cold and hot water, sparingly soluble in ethanol but insoluble in acetone and chloroform.
- The viscosity of the gum is concentration, pH and temperature dependent. The viscosity increases with increase in pH and concentration, but decreases with increasing temperature.
- The calculated apparent flow activation energy of *A. furrigunea* gum is relatively low at low concentration and reflects fewer intra and intermolecular interaction.
- The surface morphology of *A. furrigunea* gum consists of amorphous shaped molecules. The gum is more of random coil like and there is an absence of molecular entanglement within the gum.
- From the variation of speed of rotation with viscosity, viscosity with shear rate, shear stress with shear rate, it can be confirmed that *A. furrigunea* gum is a non-Newtonian fluid with characteristics shear thickening property.
- The results obtained from elemental analysis indicate that *A. furrigunea* gum is a good source of mineral nutrition.

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