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Full Length Research Paper

Physicochemical properties and rheological behaviour of *Ficus glumosa* gum in aqueous solution

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The physicochemical properties, cationic composition and rheological behaviour of exudates from *Ficus glumosa* were investigated. The gum was found to be mild acidic, ionic and soluble in water, sparingly soluble in ethanoland insoluble in acetone and chloroform. The trend for the decreasing concentration of elements determined in the gum sample is Mg > Ca > Mn > Zn > Fe > Ni > Zn > Cu > Cd > Pb. Rheological properties of the gums have been adequately modeled using Huggins, Kraemer, Arrhenius, Tanglertpaibul and Rao theories. The results indicated that the viscosity of the gum increases with increasing pH and concentration but decreases with increase in temperature. Values of intrinsic viscosity obtained from Huggins and Kraemer plots were similar (11.197). The value of activation energy (E_{vis}) of the gum studied was found to be 1.915 kJ/mol. The low E_{vis} values obtained suggests the existence of few inter and intra-interactions between the molecules of the gums within the investigated temperature. *F. glumosa* gum exudate hence, possesses the potentials to be used as an additive in food and other industries.

Key words: *Ficus glumosa*, gum, heavy metal, characterization, rheological modeling.

INTRODUCTION

Gums are considered to be pathological products formed following injury to the plant or owing to unfavourable conditions, such as drought, by a breakdown of cell walls (extra cellular formation; gummosis) (Jani et al., 2009). They are complex carbohydrate derivatives of a polysaccharide nature and are either soluble in water as in the case of gum *Arabic* or form mucilages by the absorption of large amounts of water (gum tragacanth). Their principal use is in foodstuffs by nature of their ability to impart desired qualities to foods by influencing their viscosity, body and texture; most frequently in confectionery food, flavouring and soft drinks. They also have pharmaceutical and industrial applications as demulcents, adhesives in pill manufacture, lithography, paints, inks, corrosion inhibitors and as emulsifying agents. These gums found wider application because of their physical, rheological and chemical properties (such properties include solubility, water sorption, swelling capacity, pH, effect of temperature, and viscosity among others) (De Paula et al., 2001).

There is increasing in demands for gums globally because of its vast application, which causes the increase in prices of the existing gums in the local and international market. For the price to be stable and less expensive there is need to look for alternative or discoveries of more suitable natural gums which will be of the same quality or even surpass the existing ones. *Ficus glumosa* is an example of lesser known plant producing gum. The plant commonly called African rock fig tree in English and 'kawuri' in Hausa, belongs to the Family, Moraceae. *F. glumosa,* a tall plant of about 24 m in height and about 3 m in diameter, occurs on rocky outcrops, where it splits rocks; along dry watercourses or in open country; frequently in valleys, where it reaches its greatest size (Keay, 1989). The species also occurs in fringe forest in savannah areas, especially in swampy ground, and in swamp forest in coastal areas (Arbonnier, 2004). The bark also contains abundant sticky white latex which is used in Northern Nigeria like bird-lime to trap crickets (Ameh et al., 2012). The exudate is also chewed as chewing-gum and used for fastening arrowheads to their shaft.

Studies on the physicochemical and rheological properties of some gum exudates have been carried out.

Gum exudates from *Khaya senegalensis* (Family, Meliaceae) plants grown in Northern Nigeria were investigated for its physicochemical properties such as pH, water sorption, swelling capacity and viscosities at different temperatures using standard methods by Mahmud et al. (2008). It was found that *Khaya* gum appeared to be colourless to reddish brown translucent tears. 5%w/v mucilage has pH of 4.2 at 28°C. The gum is slightly soluble in water and practically insoluble in organic solvents. Water sorption studies revealed that it absorbs water readily and is easily dehydrated in the presence of desiccants. A 5%w/v mucilage concentration gave a viscosity value which was unaffected at temperature ranges (28 to 40°C). The results indicated that the swelling ability of *K. senegalensis* gum may provide potentials for its use as a disintegrant in tablet formulation, as a hydro gel in modified release dosage forms and the rheological flow properties may also provide potentials for its use as suspending and emulsifying agents owing to its pseudo plastic and thixotropic flow patterns.

Mhinzi (2002) 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 *A. 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.

Rheological properties of Xanthan and locust beans gums have been studied by Higiro et al. (2006) and the gums were found to obey Huggins and power law models and from the rheological modeling of the gums, the conformation of the gums were established. The aim of this work therefore is, to establish some of the physicochemical and rheological properties of *F. glumosa* gum, hence providing the information about the nutritional values, the metal composition and the functional properties of the gum.

MATERIALS AND METHODS

Collection of samples

Crude *F. glumosa* gum was obtained as dried exudates from their parent trees grown at Adepe - Otukpain Ogbadibo LGA of Benue State, Nigeria. The plant material had earlier been identified and authenticated in the herbarium Department of Biological Sciences of Ahmadu Bello University Zaria. The gum were collected from the plant species by tapping during the mid of July and in the day time (Smith and Montgomery, 1959).

Purification of the gum

The crude sample of *F. glumosa* 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 1 to 2 weeks.

The crude gum was dissolved in cold distilled water and the solution was strained through muslin and was then centrifuged, depositing a small 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%. 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.

Physiochemical analysis

In order to characterize the gums, it was subjected to the following physiochemical tests:

Determination of 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.

Determination of solubility

The solubility of the gum was determined in cold and hot distilled water, acetone, chloroform and ethanol. 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 (Model.XP-3000) and expressed as the percentage solubility of the gums in the solvents (Carter, 2005).

Determination of concentration of metals

Concentrations of Zn, Mg, Ca, Mg, Mn, Fe, Cu, Cd and Pb were determined using perkin Elmer atomic absorption spectrophotometer. Calibration curve for each metal was prepared and the concentration of the metal was in the analyte and was estimated by extrapolation.

Determination of nitrogen and protein content

Crude protein content of the gum was determined using the Kjeldahl method with the nitrogen content being multiplied by a factor of 6.25 (Rodriguez et al., 2004).

Determination of pH

The sample powder was thoroughly mixed and 1 g and 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 (Oaklon pH meter, Model 1100).

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

Table 1. Physiochemical parameters of *F. glumosa* gum.

Parameter	*F. glumosa* gum
Colour	Pale – yellow
Odour	Honey
Taste	Bland
pH	4.3
Percentage yield (% w/w)	68%
Solubility (% w/v)	
Cold water	10.20
Hot water	11.10
Acetone	0.0
Chloroform	0.0
Ethanol	0.4
Nitrogen (%)	0.41
Protein (%)	2.56

Table 2. Cationic composition of *F. glumosa* gum.

Element	*F. glumosa* gum
Mg (%w/w)	1.28
Ca (%w/w)	0.60
Zn (ppm)	18.27
Mn (ppm)	105.53
Fe (ppm)	14.57
Ni (ppm)	10.12
Cu (ppm)	7.32
Cd (ppm)	1.69
Pb (ppm)	1.20

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 stirring 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 ± 0.1°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 (η_{rel}) was calculated using Equation 1:

$$\eta_{rel} = \frac{T - T_o}{T_o} \qquad (1)$$

WhereT 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.

RESULTS AND DISCUSSION

Physiochemical parameters of *F. glumosa* gum

Table 1 presents physiochemical parameters of *F. glumosa* gum. The colour of crude *F. glumosa* gum is pale yellow. After purification, the yield of gum obtained was 68%w/w. The purified gum had bland taste and is odourless. The measured pH of the gum is 4.3 indicating that the gum is mild acidic. This compared favourably with that of gum *Arabic* which is reported to be between 4.0 and 4.8 (Belitz et al., 2004). The solubility of *F. glumosa* in cold water and hot water are 10.20 and 11.10%, respectively indicating that the solubility of the gum is temperature dependent. However, since solubility is expected to increase with increase in temperature, the solubility of the gum in hot water is higher than the corresponding solubility in cold water. On the other hand, the samples were not soluble in acetone and chloroform but were sparingly soluble in ethanol. These indicate that the studied gum is ionic in character, unlike the covalent counterpart, that is soluble in organic solvents (Sarah et al., 1998). The sparingly solubility of the gum observed in ethanol, is due to the fact that it can ionize to produce hydroxyl ion (OH^-) and the value of its dielectric constant is higher than those of acetone and chloroform. Consequently, ethanol has some polar character over acetone and chloroform which are characterized by low values of dielectric constant.

The nitrogen content of *F. glumosa* gum was found to be 0.41% which is higher than *Karaya* - 0.20% but lower than that of kondagogu gum 1.0% (Janaki and Sashidhar, 1998) and *Acacia* gum - 0.44% (Mhinzi and Mrosso, 1995). Protein content of *F. glumosa* gum was found to be 2.56%. This indicates a possibility of its usage as a supplement to other protein sources in the food.

Cationic composition of *F. glumosa* gum

Table 2 presents concentration of elements identified in the gum sample. Cationic composition of *F. glumosa* gum exudate demonstrated that calcium and magnesium are the main metals present among the metals determined. These major metals are also present in *Acacia senegal* gum (Anderson and Douglas, 1988). The high level of the magnesium and calcium shows that the exudates could provide an alternative source of magnesium and calcium in diets. Magnesium is required in large quantities by the body for the activation of enzymes involved in protein synthesis. Possible outcomes of deficiency are growth failure, behavioral disturbances, weakness and spasms (Murray et al., 2000).

The trend for the decreasing concentration of other elements determined in the gum sample is Mn > Zn > Fe > Ni > Zn > Cu > Cd > Pb. The body requires in a minute quantity of Mn for the activation of various enzymes, Co is nutritionally Co^{2+} and can function as a replacement

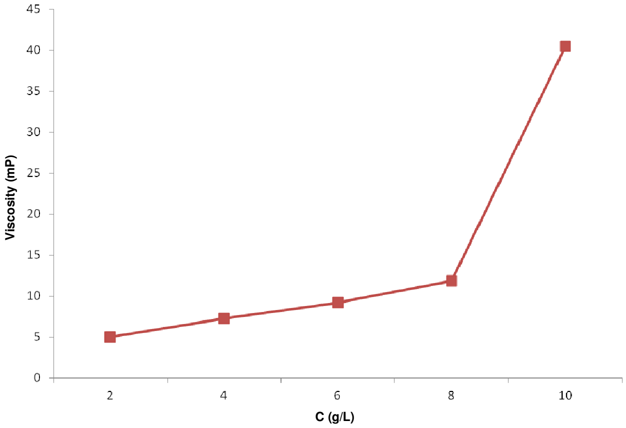

Figure 1. Variation of viscosity with concentration of *F. glumosa*, gums.

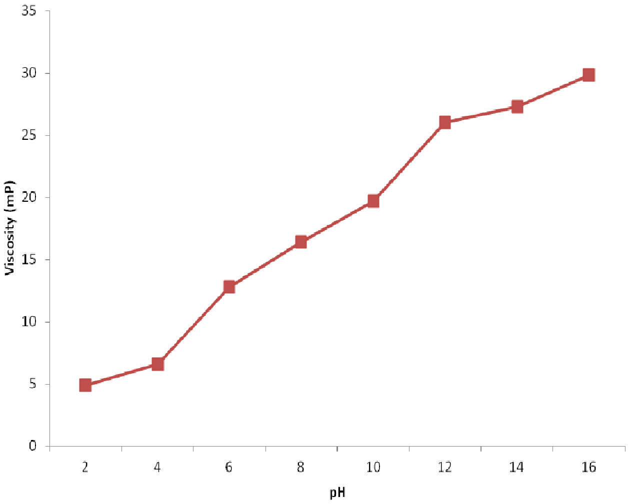

Figure 2. Variation of viscosity with pH for *F. glumosa* gum.

in vitro for other divalent cations in human (Underwood, 1977). Fe is needed in trace amount by the body as it is a constituent of hemoglobin and enzymes involved in energy metabolism in iron transport (Malhotra, 1998). The concentration of Pb detected in the gum was found to be within the permissible range of 2 ppm for substances used as food additives (FAO, 1999).

Rheological study

Rheological measurements provide useful behavioural and predictive information for various products in addition to knowledge of the effects of processing, formulation changes, and aging phenomena (Ebewele, 2000). Rheology deals with those properties of materials that determine their response to mechanical force. For solids, rheological studies involve elasticity and plasticity. For fluids, on the other hand, this involves viscosity measurements, viscosity being a measure of the internal friction of a fluid.

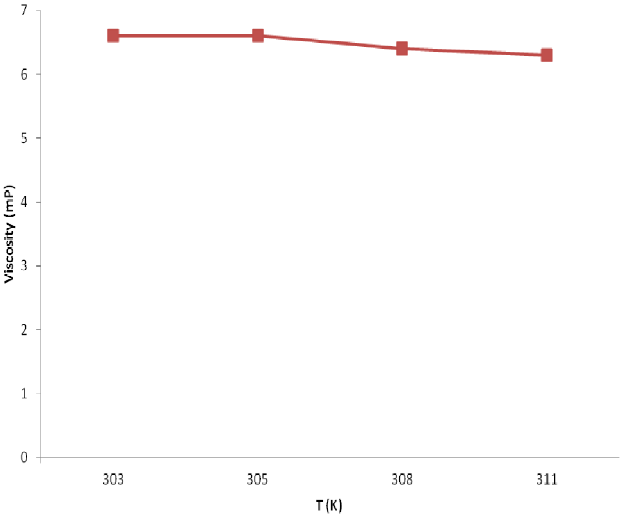

Figure 3. Variation of viscosity with temperature for *F. glumosa* gum.

Anderson and Dea (1967) stated that viscosity is one of the most important analytical and commercial parameters in polymers because it is affected by the size and shape of macro-molecules.

Figure 1 shows plots for the variation of viscosity with concentration of *F. glumosa* gum. From Figure 1, it is evident that the viscosity of *F. glumosa* gum tends to increase with increase in concentration. Jefferies et al. (1977) and Ashton et al. (1975) have reported that the viscosity of gum *Ghatti* and *Albizia* gums increases with increasing proportion of dispersible gel. This rheological behaviour is also exhibited by gum exudate studied. The high viscosity experience at higher concentrations may be due to increase in the strength of molecules-molecules interaction and the corresponding reduction in molecule-solvent interaction.

Figure 2 presents plots for the variation of viscosities of the gums with pH. Figure 2 reveals that the viscosity of the gum tend to increase with increase in pH indicating that the emulsifying properties of the gum is pH-dependent (Calvo et al., 1998).

In Figure 3, plot for the variation of viscosity with temperature for the studied gum is presented. In order to verify the onset of degradation or conformational transitions during heating, the viscosity of the gums was again measured as cooling proceeded. The tests did not indicate any difference between the two set of data suggesting that there was no degradation or conformational transition when the gums were heated (De Paula et al., 2001). Figure 3 revealed that the viscosity of the gum decreases with increasing temperature. This trend occurs because the increased kinetic motion at higher temperatures promotes the

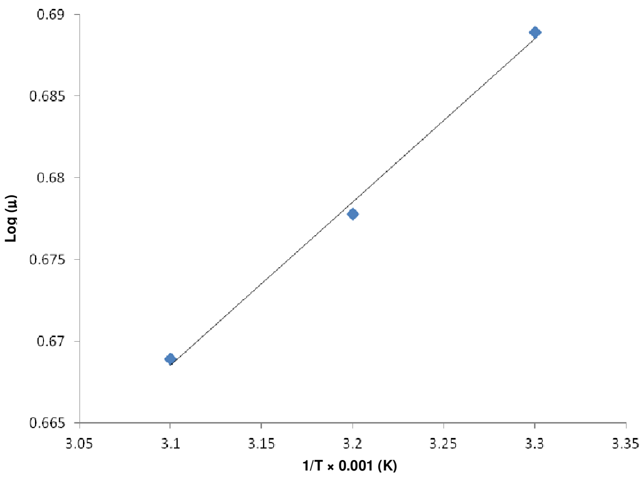

Figure 4. Arrhenius molecular kinetic plots of *F. glumosa* gum.

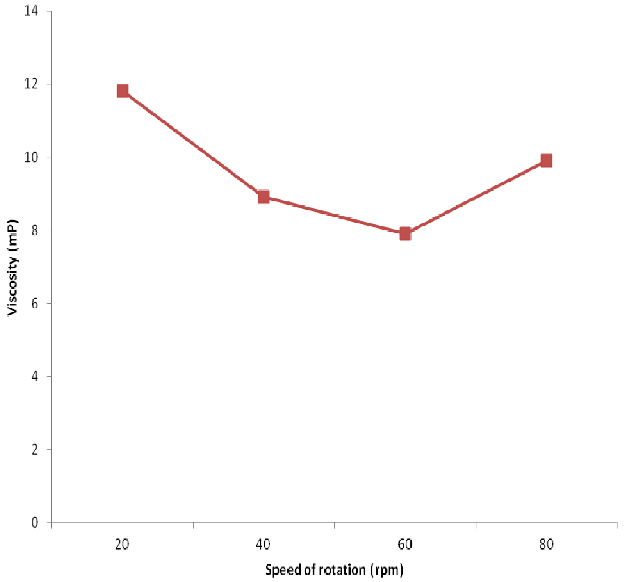

Figure 5. Variation of viscosity with rotation speed of *F. glumosa* gum.

breaking of intermolecular bonds between adjacent layers. From the viscosity results, the activation energy of viscous flow, E_{vis}, of the gum exudate was determined using the Arrhenius equation given by Equation 2:

$$[\eta] = Ae - E_{vis} / RT \qquad (2)$$

Where T is temperature, A is the Arrhenius coefficient and R is the universal gas constant. A plot of log $[\eta]$ against I/T was made. The results obtained indicated a linear ($R^2 = 0.9959$) relationship between log(η) and 1/T. The slope of the plot (Figure 4) is equal to $-E_{vis} / 2.303R$ from which E_{vis} was evaluated. The value of E_{vis} of the gum studied was found to be 1.915 kJ/mol. The calculated activation energy of flow is relatively low when compared with data obtained for some plant gums such as *Arabic* gum (15 kJ/mol) (Chikamai and Banks, 1993) and *Albizia lebbeck* gum (15.9 kJ/mol) (De Paula et al., 2001). The low E_{vis} values obtained suggests the existence of few inter and intra-interactions between the molecules of the gums within the investigated temperature.

In order to study the effect of centrifugal forces on the viscosity of the gum, the viscosity of the gum were measured at various speeds of rotation. Figure 5 shows the plot of viscosity versus the rotation speed of a centrifuge containing similar concentration of the gum. From Figure 5, 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 increases with increase in speed of rotation. 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 (2003) 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. Therefore, alteration in the composition of the various gums as a result of rotation may be responsible for the observed changes in viscosity of the gums with rotation speed of the centrifuge.

Intrinsic viscosity [η], represents the volume occupied by the unit mass of the polymer molecule. It is not actually a viscosity. This parameter is generally measured in order to obtain information about the molecular weight and conformation of a polymer (Hill et al., 1998; Ross-Murphy, 1995). Intrinsic viscosity is determined experimentally by measurements of the viscosity of solutions of very low concentrations (c<c*). Denoting solution and solvent viscosity as, η and η_s, respectively, it is defined formally by the following standard relationships:

$$\text{Relative viscosity: } \eta_{rel} = \eta / \eta_s \qquad (3)$$

$$\text{Specific viscosity: } \eta_{sp} = (\eta - \eta_s) / \eta_s = \eta_{rel} - 1 \qquad (4)$$

$$\text{Intrinsic viscosity: } [\eta] = \lim_{c \to 0} \frac{\eta_{sp}}{C} \qquad (5)$$

The intrinsic viscosity [η] was determined using Huggins, Kraemer Tanglertpaibul and Rao plots as proposed by Higiro et al. (2006).

The Huggins equation is given as Equation 6:

$$\eta_{sp} / c = [\eta] + K' [\eta]^2 C \qquad (6)$$

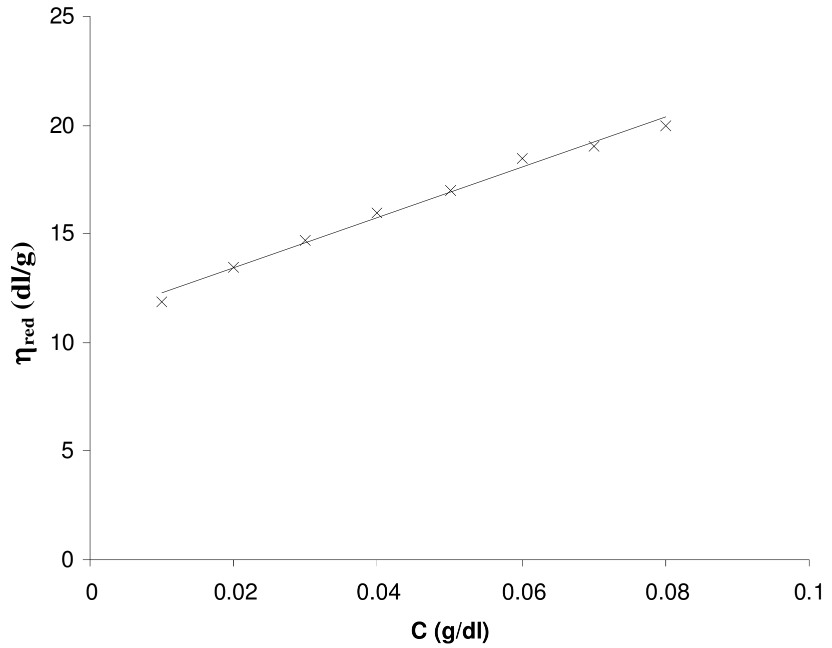

Figure 6. Variation of reduced viscosity with concentration (Huggins plot) for *F. glumosa* gum.

Table 3. Huggins and Kraemer parameters for *F. glumosa* gum.

Plot	Slope	[η]	$[\eta]^2$	k	R^2
Huggins	114.32	11.197	120.734	0.955	0.9874
Kraemer	8.300	11.397	136.306	0.059	0.9544

It can be deduced from Equation 6 that a plot of η_{red} versus C should be linear with slope equal to $[\eta]^2$ and intercept equal to [η]. Figure 6 shows Huggins plot for the gum exudate. Values of Huggins constant deduced from the plot are presented in Table 3. Based on experimental observations, the physical meaning of the dimensionless Huggins coefficient k can be summarized as follows (Sakai, 1968):

1) A polymer exhibits a higher value of k in a poor solvent than in a good solvent, that is, when the polymer-polymer interactions become favourable over polymer-solvent interactions,
2) It has a value of 0.5 to 0.7 in a theta solvent,
3) k is very sensitive to the formation of molecular aggregates.

The value of k obtained for the studied sample is 0.955. Therefore, the degree of polymer-polymer interaction in *F. glumosa* gum is low.

The intrinsic viscosity was also analyzed using the Kraemer equation, which is given by Equation 7 (Higiro et al., 2006) as follows:

$$\ln(\eta_{rel}/C) = [\eta] + k\ [\eta]^2 C \qquad (7)$$

Where k″ is the Kraemer constant. From Equation 7, a plot of $\ln(\eta_{rel}/C)$ versus C should be a straight line with intercept and slope equal to [η] and k″[η], respectively. Figure 7 shows Kraemer plots for the studied gum. Values of Kraemer parameters deduced from the plot are recorded in Table 3. From the results obtained, k″values obtained from Kraemer plot is comparable to k' values obtained from Huggins plot. However, value of [η] deduced from Huggins plots are relatively higher than those obtained from Kraemer plot.

Tanglertpaibul and Rao (1987) obtained three equations that can also be used in the determination of the intrinsic viscosity of a polymer. These are Equations 8, 9 and 10.

$$\eta_{rel} = 1 + [\eta]C \qquad (8)$$

$$\eta_{rel} = \exp([\eta]C) \qquad (9)$$

$$\eta_{rel} = 1/(1-[\eta]C \qquad (10)$$

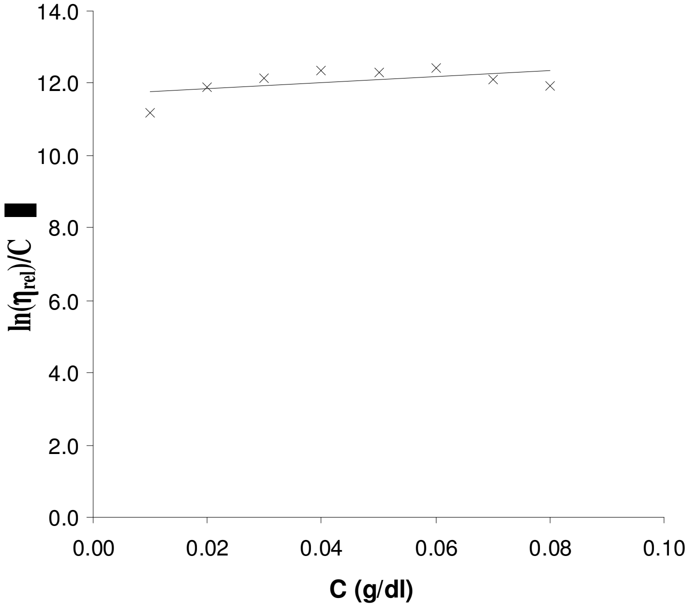

Figure 7. Variation of ln(relative viscosity) with concentration (Kraemer plot) of *F. glumosa* gum.

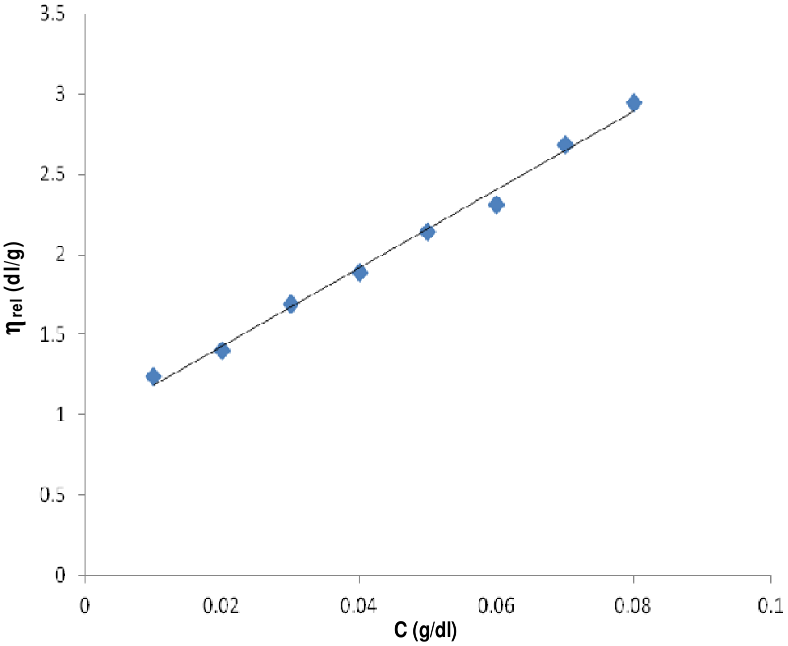

Figure 8. Variation of η_{rel} with concentration of *F. glumosa* gum.

From Equation 8, [η] is the slope obtained by plotting η_{rel} versus C. Also, the implication of Equation 9 is that, [η] is the slope obtained from the plot of $\ln\eta_{rel}$ versus C and from Equation 10, [η] is a slope obtained by plotting (1-$1/\eta_{rel}$) versus C. Tanglertpaibul and Rao plots for the studied gum (from Equations 8 to 10) are presented in Figures 8 to 10, respectively. Values of Tanglertpaibul and Rao parameters deduced from the plots are presented in Table 4. The excellent correlation between the values of [η] computed from the various methods

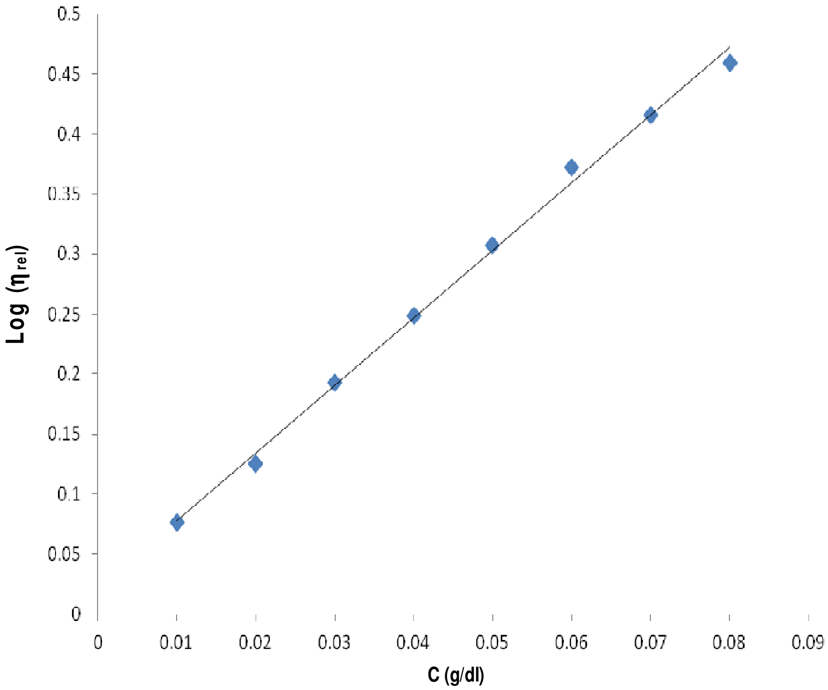

Figure 9. Variation of Log(η_{rel}) with concentration of *F. glumosa* gum.

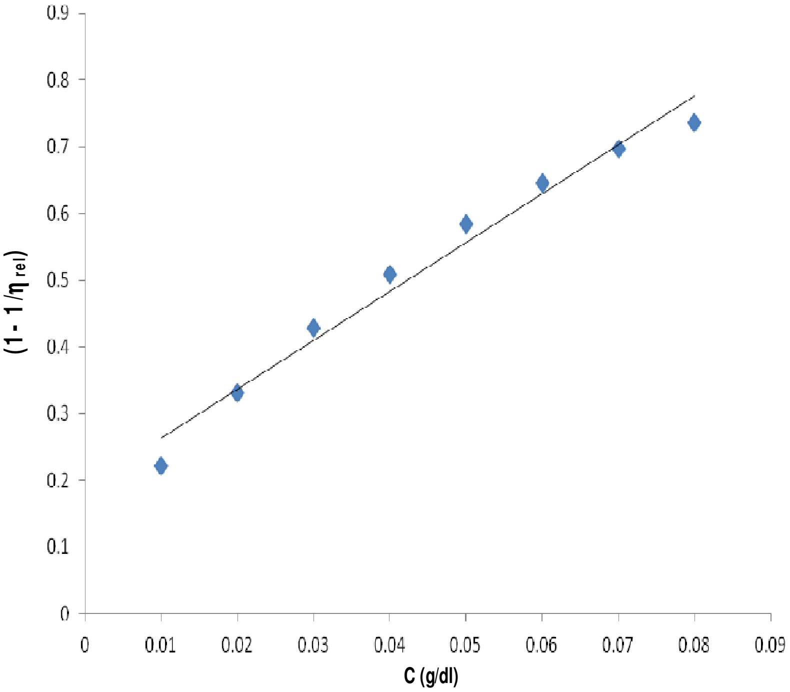

Figure 10. Variation of (1- 1/η_{rel}) with concentration of *F. glumosa* gum.

used in evaluating intrinsic viscosity indicates the consistency of the methods for the study of intrinsic viscosity of the system.

Conclusions

From the results obtained from the present study, the

Table 4. Tanglertpaibul and Rao parameters for *F. glumosa* gum.

Gum	Equation 8		Equation 9		Equation 10	
	[η]	R^2	[η]	R^2	[η]	R^2
F. glumosa	24.40	0.9928	5.6254	0.9967	7.3244	0.9756

following conclusions are drawn:

1) *F. glumosa* gum occurs as pale yellow, translucent tears. The gum mucilage has a bland taste, odourless, and mild acidic with a pH of 4.3 at 303 K. It is soluble in water, sparingly soluble in ethanol and practically insoluble acetone and chloroform.
2) The mineral elements compositions of the gum are within the range specified by World Health Organization (WHO) and Food and Agriculture Organization (FAO). Hence, *F. glumosa* gum exudate can be used as an additive in food and other industries.
3) *F. glumosa* gum is a macromolecule whose viscosity depends on temperature, concentration, speed of rotation and pH.
4) The rheological properties of the gum can be adequately modeled using the Arrhenius model, Huggins model, Kraemer model, Tanglertpaibul and Rao models. The excellent correlation between the values of [η] computed from the various methods used in evaluating intrinsic viscosity indicates the consistency of the methods for the study of intrinsic viscosity of the system.

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