



Determination of Intrinsic Viscosity and Molecular Weight of Xanthan Gum by Plots of Different Equations

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ABSTRACT

Xanthan gum (XG) is a natural high molecular weight polymer. It is one of the important polysaccharides, produced by the fermentation process. This research aims to estimate the intrinsic viscosity of XG. The intrinsic viscosity $[\eta]$ was evaluated by various equations, initially by Huggins equation as a reference, then using other equations, it was compared and calculated with the relative percentage of errors. From the calculations, we note that the Arrhenius-Rother-Hoffmann is the most reliable method for estimating the intrinsic viscosity, while the Fuoss method is very unsuitable for estimating the intrinsic viscosity. The fundamental importance of intrinsic viscosity lies in the fact that it's the first step to estimating the molecular weight (1043107 g/mol) and hydrodynamic radius (280 nm). Furthermore, the value of molar mass was calculated by the Mark-Houwink equation for helical conformation with parameters $a = 0.74 \times 10^{-3} \text{ cm}^3/\text{g}$ and $k = 1.35$. where a and k are M-H constants depending upon the type of polymer.

Keywords:

Huggins equation - Intrinsic – Viscosity - Mark-Houwink equation - molecular weight - Xanthan gum

تقدير اللزوجة الجوهريّة والوزن الجزيئي لصبغ الاكزنتان بواسطة الرسم البياني لمعادلات مختلفة

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الملخص

صبغ الاكزنتان هو بوليم طبيعي له وزن جزيئي عالى وهو احد اهم المركبات عديدة السكر، ينتج بواسطة عملية التخمر. الهدف من هذا البحث هو حساب اللزوجة الجوهريّة لصبغ الاكزنتان. اللزوجة الجوهريّة $[\eta]$ تقدر بواسطة معادلات مختلفة، ابتداءً من معادلة هيجنس والتي تعتبر كمعادلة مرجعية، ثم تستخدم معادلات اخرى لحساب اللزوجة الجوهريّة ويتم مقارنتها وحسابها مع نسبة مئوية للخطأ. من الحسابات لاحظنا ان طريقة ارهينيوس-رزفورد-هوفمان هي اكثر

الطرق الموثوقة لحساب اللزوجة الجوهريّة، بينما طريقة فيوز غير ملائمة جدا لحساب اللزوجة الجوهريّة. الأهمية الأساسية من حساب اللزوجة الجوهريّة تكمن في كونها الخطوة الأولى لحساب الوزن الجزيئي (1043107 جم/مول) ونصف القطر الهيدروديناميكي (280nm). بالإضافة لذلك قيمة الكتلة المولارية حسبت بواسطة معادلة مارك-يونيك.

الكلمات المفتاحية: اللزوجة الجوهريّة، الوزن الجزيئي، صمغ الاكزنتان، معادلة هيجنس، معادلة مارك يونيك

Introduction

Xanthan gum is a microbial polysaccharide with many industrial uses. It is an effective thickening agent and stabilizer to prevent ingredients from separating, it can be produced from aerobic fermentation of the bacterium *Xanthomonas campestris* [1,2]. The chemical formula of its monomer (C₃₅H₄₉O₂₉), with a molar mass of 933.748 gmol⁻¹ [3]. Xanthan, which consists of pentasaccharides repeating units with β-1,4 glycosidic linked glucose molecules that are similar to cellulose as the main chain, (Figure 1) is completely soluble in water[3]. Xanthan gum shows an order confirmation, double-stranded helical structure, and disorder conformation, as a single-standard random coil [3]

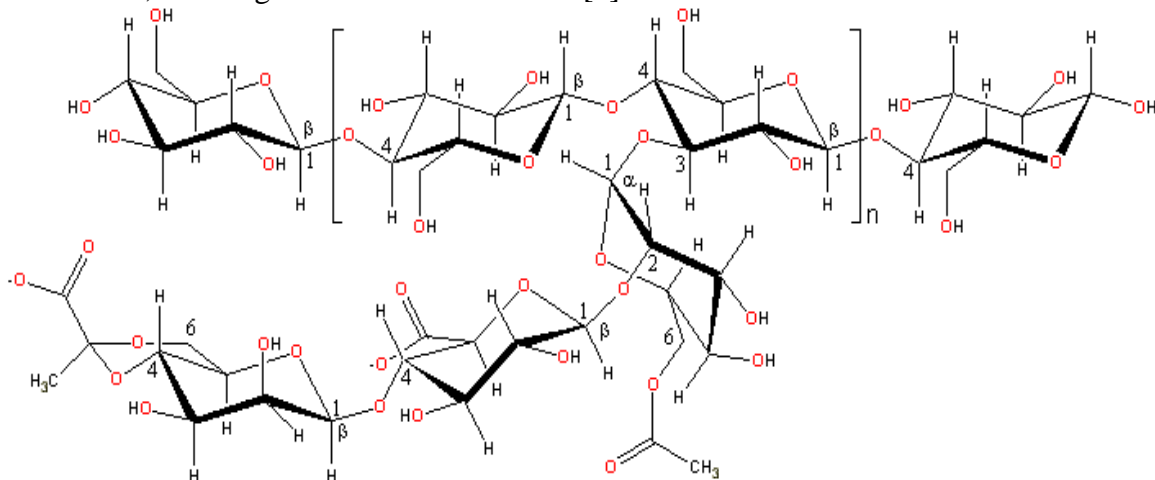


Figure 1. Structure of monomer of xanthan gum

Xanthan gum is highly soluble and viscous even at high dilutions [4,5], stable over a wide range of temperatures and pH, hence xanthan gum is widely used in pharmaceutical, cosmetics, agriculture and petroleum industries, food, cosmetics industries, and as a thickener drilling oil and toothpaste [5].

also, xanthan gum exhibits very high molecular based on many previous studies[3-5]

1.1 Intrinsic Viscosity and Hydrodynamics Parameters

The viscometer was used to measure the efflux time of polymer solution and solvent (water).

The relative viscosity is measured by a simple relation



$$\eta_r = \frac{\eta_s}{\eta_o}$$

$$= \frac{\rho_s \cdot t_s}{\rho_o \cdot t_o}$$

Where; η_s is the solution viscosity, η_o is the water viscosity, ρ_s and ρ_o are densities of solution and water respectively, t_s and t_o are efflux time of solution and water, respectively[6].

And the specific viscosity η_{sp} measured as

$$\eta_{sp}$$

$$= \eta_r$$

$$- 1$$

And reduced viscosity η_{red} measured as

$$\eta_{red}(cm^3 g^{-1})$$

$$= \frac{\eta_{sp}}{C} \quad (3)$$

Where, C is the concentration.

Also, inherent viscosity η_{inh} measured from the following relation[6]

$$\eta_{inh}(cm^3 g^{-1})$$

$$= \frac{\ln \eta_r}{C} \quad (4)$$

Huggins [6], propose the following equation

$$\eta_{red}$$

$$= [\eta]$$

$$+ K_H [\eta]^2 C$$

Where, $[\eta]$ intrinsic viscosity, and K_H is Huggins constant

The Kraemer [7], propose the following equation

$$\eta_{inh} =$$

$$[\eta] +$$

$$K_K [\eta]^2 C \quad (6)$$

Where, K_K is Kraemer constant

Fuoss [8], propose the following equation

$$\frac{C}{\eta_{sp}}$$

$$= \frac{1}{[\eta]}$$

$$+ K_{FS} \cdot \frac{1}{[\eta]} \cdot C^{1/2}$$

Where, K_{FS} is Fuoss constant

Martin [9], propose the following equation



$$\begin{aligned} & \ln \eta_{red} \\ & = \ln \ln [\eta] \\ & + K_M [\eta] C \end{aligned} \quad (8)$$

Where, K_M is Martin constant

The Fedors [10] propose the following equation:

$$\begin{aligned} & \frac{1}{2(\eta_r^2 - 1)} \\ & = \frac{1}{[\eta]c} \\ & - \frac{1}{[\eta]C_{max}} \end{aligned} \quad (9)$$

Where C_{max} is polymer concentration parameter.

Heller [11] propose the following equation :

$$\begin{aligned} & \frac{1}{2} c \left(\frac{1}{\eta_{sp}} + \frac{1}{\ln \eta_r} \right) \\ & = \frac{1}{[\eta]} \\ & - K_{He} c \end{aligned} \quad (10)$$

Where K_{He} is Heller's constant.

Lyons and Tobolsky [12] propose the following equation :

$$\begin{aligned} & \ln \frac{\eta_{sp}}{c} \\ & = \ln \ln [\eta] \\ & + \left(\frac{K_{L-T} [\eta] c}{1 - bc} \right) \end{aligned} \quad (11)$$

Where K_{L-T} is Lyons and Tobolsky constant , b is constant

Baker [13] propose the following equation:

$$\begin{aligned} & \frac{1}{\eta_r^n} \\ & = 1 \\ & + [\eta] \frac{c}{n} \\ & = \frac{1}{1 - 2K_H} \end{aligned}$$

With $0.25 < K_H < 1$, and $K_H = 0.48$

Tager [14] propose the following equation:



$$\begin{aligned} & \frac{C}{\eta_{sp}} \\ &= \frac{1}{[\eta]} (K_{T1} \\ &+ K_{T2}C^2) \end{aligned} \quad (14)$$

Where K_{T1} and K_{T2} are Tager's constants

Budtov [15] propose the following equation:

$$\begin{aligned} & \eta_r^{K_B} \\ &= 1 \\ &+ K_B[\eta]c \end{aligned} \quad (15)$$

$$\begin{aligned} & K_B \\ &= 1 \\ &- 2K_M \end{aligned} \quad (16)$$

Where K_M is Martin's constant. This method used to determine the intrinsic viscosity when $K_M > 0.5$.

Solomon & Gotesman [16] propose the following equation :

$$\begin{aligned} & 1 + \frac{1}{3}\eta_{sp} \\ &= [\eta] \frac{\eta_{sp}}{c} \end{aligned} \quad (17)$$

Arrhenius-Rother-Hoffmann [17,18] propose the following equation:

$$\begin{aligned} & \frac{\ln \eta_r}{c} \\ &= [\eta] \\ &+ K_A \ln \eta_r \end{aligned} \quad (18)$$

Where K_A is constant.

Kresia [19] propose the following equation:

$$\begin{aligned} & \frac{\eta_{sp}}{c} \\ &= [\eta] \\ &+ K_{Kr} \frac{\eta_{sp}^2}{c} \end{aligned} \quad (19)$$

Where K_{Kr} Kresia's constant.

Staudinger and Heuer [20] propose the following equation:

$$\begin{aligned} & \ln \frac{\eta_{sp}}{c} \\ &= \ln [\eta] \\ &+ k_{S-H}[\eta]c \end{aligned} \quad (20)$$



Where K_{S-H} is Staudinger - Heuer constant.

Maron & Reznik [21] propose the following equation:

$$\begin{aligned} & \frac{\eta_{sp} - \ln\eta_r}{c^2} \\ &= \frac{[\eta]^2}{2} \\ &+ \left(k_H \right. \\ &\left. - \frac{1}{3} \right) [\eta]^3 c \end{aligned} \quad (21)$$

By modifying Maron –Reznik equation

$$\begin{aligned} & \ln \left(\frac{\eta_{sp} - \ln\eta_r}{c^2} \right) \\ &= \ln \left(\frac{[\eta]^2}{2} \right) + \ln \left(\left[k_H - \frac{1}{3} \right] [\eta]^3 \right) \\ &\times \ln c \end{aligned} \quad (22)$$

In this research two alternatives are proposed to determine the intrinsic viscosity. The first method is a square method (S), through which we can estimate the intrinsic viscosity from the intercept of a straight line at drawing equation 23.

$$\begin{aligned} & \left(\frac{\eta_{sp}}{c} \right)^2 \\ &= [\eta]^2 \\ &+ k_{P2} c^2 \end{aligned} \quad (23)$$

The relative percentage of error (RE%) with respect to Huggins of this equation is by default.

The second method is the square root method (SR), in which we can calculate the intrinsic viscosity from the intercept of a straight line when drawing equation 24.

$$\begin{aligned} & \left(\frac{\eta_{sp}}{c} \right)^{1/2} \\ &= [\eta]^{1/2} \\ &+ k_{P1} c^{\frac{1}{2}} \end{aligned} \quad (24)$$

Where, k_p is square method constant.

The percentage relative error (RE%) with respect to Huggins equation is neglected.

In addition, the average of both intrinsic viscosities gives a value not exceeding 5% regarding the Huggins method.

From Mark-Houwink (M-H) [22,23] equation (25) we can calculate the molar mass given the value of intrinsic viscosity. Since the size of the polymer chain is related to the molar mass[22,23].



$$[\eta] = kM_w^a \quad (25)$$

Mark-Houwink parameters are calculated by using the plot of the following equation:

$$\ln [\eta] = \ln k + a \ln M_w \quad (26)$$

Where k is M-H proportionality constant and a is M-H exponential constant, is a function of polymer geometry and takes values from 0.5 to 2.0 . The M-H exponent represents the imprint of the polymer chains inside the solution.

The hydrodynamic radius (R_H) is calculated from Einstein relation

$$M[\eta] = v_a N_A \frac{3}{4} \pi (R_H)^3$$

The relation between intrinsic viscosity and specific volume is given by the following equation [24,25]:

$$\frac{v_{a/b}}{V_s} = \frac{[\eta]}{V_s}$$

Where $v_{a/b}$ is called Einstein viscosity increment and V_s is specific volume (cm^3/g) [26].

2. Experimental

2. 1. Material and Mother Solution:

The powder material of xanthan gum was supplied by a local Libyan company (JOWF) and used without further purification. The mother solution of 1g/L concentration was prepared by adding a known weight of the polymer to a fixed volume of double distilled water and dissolving with magnetic stirring. Some extra double distilled water was, then added up to the required volume.

2. 2. Dilute Solutions:

The native solution, was used to prepare a series of dilute solutions (0.1 to 0.9 gL^{-1}), by adding the required volumes of double distilled water to different volumes of native solution. A centrifuge was used at 2000 rpm for 15 min to remove bubbles from the solutions.

2. 3. Density Measurements:

The densities of solutions were measured by density bottle at 25°C .

2. 4. Viscosity Measurements:

Values of efflux time of solutions were measured in the thermostatic bath by Ostwald glass capillary viscometer (No. II: 75-100 s, Nach, Ostwald 486510, made by the brand in Germany), with water efflux time 78.32 s. The accuracy of the viscosity measurement was ± 0.015 . All the measurements were performed in triplicate.

3. Results & Discussion



Diluted solutions of xanthan gum were prepared and the intrinsic viscosity was determined by different methods. From the data obtained by using the Huggins method, the intrinsic viscosity was calculated with a value of $98622.4 \text{ cm}^3/\text{g}$.

Figs. 2 and 3 show the data obtained from the Huggins and Kraemer methods, these Figures illustrate the difficulty of both methods converging for the calculation of the intrinsic viscosity, the Huggins method was taken as standard and then, the other methods are compared with it. The intrinsic viscosity data are compared in Table (1).

Fig. 4 shows the application of Martin's method from this Figure observed, the calculation of the intrinsic viscosity accounts for an (RE%) of 2.71%, but $R^2=0.97$; in similar works such as Martin A. Masuelli [24].

Fig. 5 and Fig. 6 are the methods of Fuoss and Fedors, each method has become more pertinent in the last years, in the first RE% is 8.1%, and the second less than 1%.

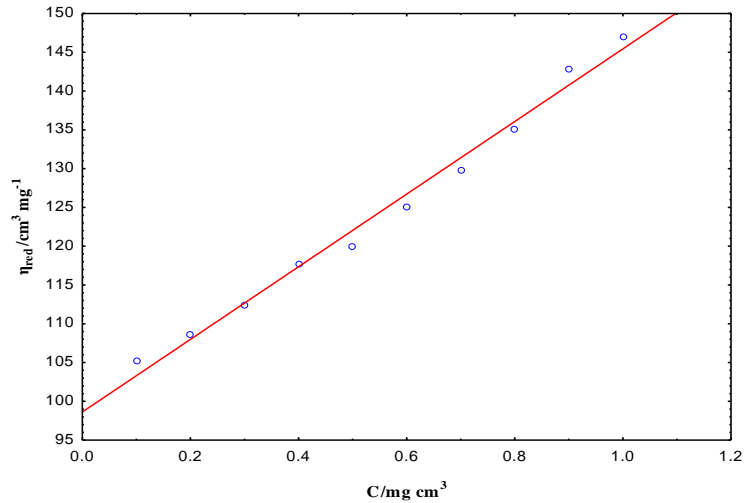
However, the method of Heller in Figure 7, is one of the most methods used in the last years this method obtains a RE% close to 3 which makes it suitable for this type of macromolecules.

Fig. 8 shows the Lyons-Tobolsky method that stands out for its RE% less than 2% that can be very feasible at the time of using a suitable plot method of calculating the intrinsic viscosity.

Moreover, Fig. 9 shows the Baker method, this method has been reliant on the Huggins method since in this case n contains the Huggins constant, KH , and not only carries the errors of the Huggins method but adds its own, which leads to RE% 3.6. Therefore, Baker's method is insufficient if one wants to determine the intrinsic viscosity. Fig. 10 shows the Tager method, which has a RE% of 1.63. The Solomon Gotesman method, Fig. 11, is an empirical equation that provides intrinsic viscosity data with RE% 0.71%, which makes it a very suitable method for this type of macromolecules. The Arrhenius-Rother-Hoffmann equation seen in Fig. 12, is one of the most accurate methods of graphical calculation of the intrinsic viscosity with RE% 0.5. This classic method is worldwide and appropriate for macromolecules of all types, although it is not widely used. Fig. 13 observed the use of the Kreisa method for the plot calculation of the intrinsic viscosity with a RE% of 3.38%. However, Staudinger-Heuer in Fig. 14 with a RE% of 3.16. The Maron-Reznik method,



Figure 15, has RE% 2.39). Buttons. method, Figure 16, is a slope model of Martin's constant,



KM, which, RE%=1.73..

Figure 2 Huggins method

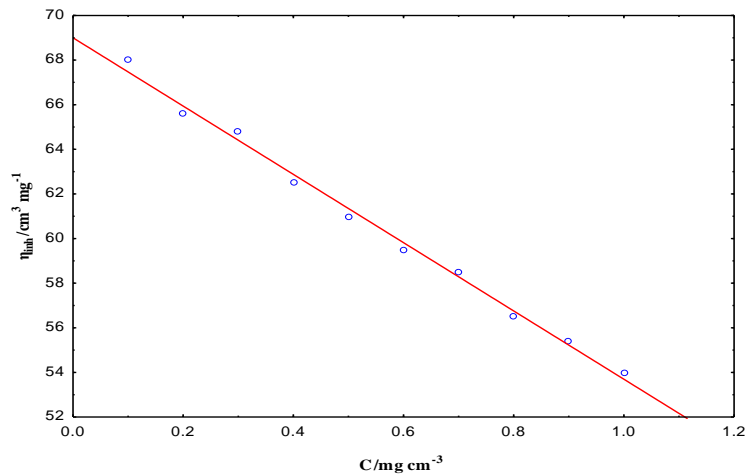


Figure 3 kraemer method

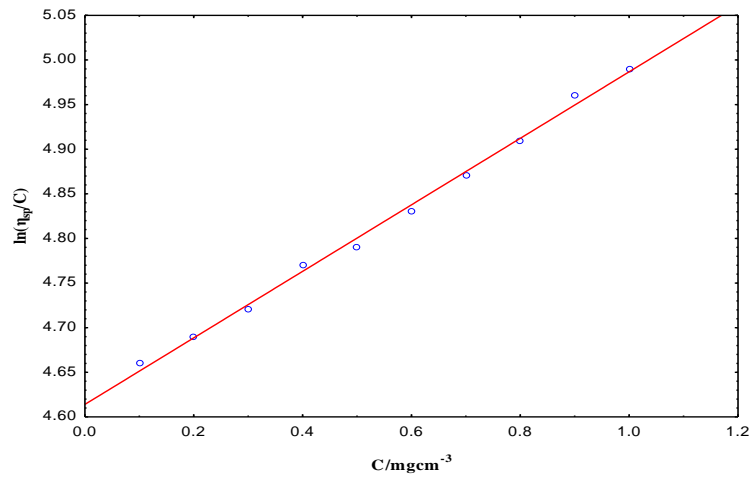


Figure 4 Martin's method

Table 1. The intrinsic viscosity by different methods

Methods	Huggins	Kraemer	Fuoss	Martin	Fedors
$[\eta]$ (cm ³ /g)	98622.4	93452.3	90632.6	95948.4	97778.2
R²	0.97	0.98	0.98	0.97	0.99
%RE	-	5.24	8.10	2.71	0.85
Methods	Heller	Lyons-Tobolsky	Baker	Tager	Budtov
$[\eta]$ (cm ³ /g)	101368.9	96652.11	94984.21	97005.41	100332.23
R²	0.98	0.99	0.87	0.95	0.94
%RE	2.78	1.97	3.6	1.63	1.73
Methods	Solomon-Gotesman	Arrhenius-Rother-Hoffmann	Kreisa	Staudinger-Heuer	Maron-Reznik
$[\eta]$ (cm ³ /g)	99325.3	98125.4	101960.3	101742.2	100984.2
R²	0.92	0.94	0.95	0.98	0.93
%RE	0.71	0.50	3.38	3.16	2.39
Methods	Square	Square Root	Mean		
$[\eta]$ (cm ³ /g)	103997.5	95423.1	99710.3		
R²	0.97	0.94	-		
%RE	5.45	2.10	1.10		

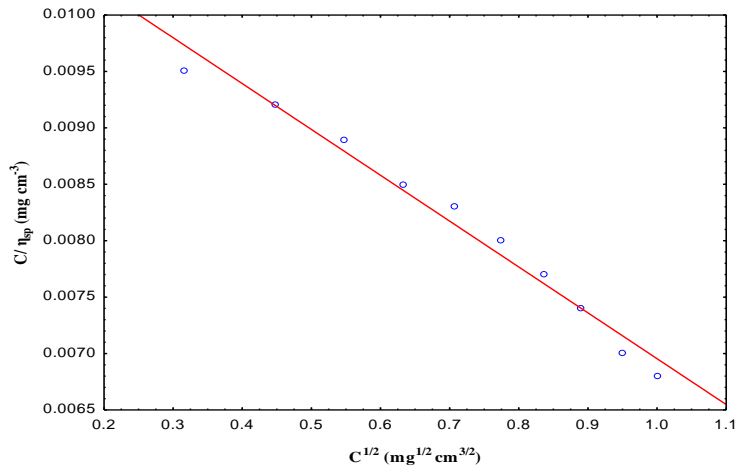


Figure 5 Fuoss method

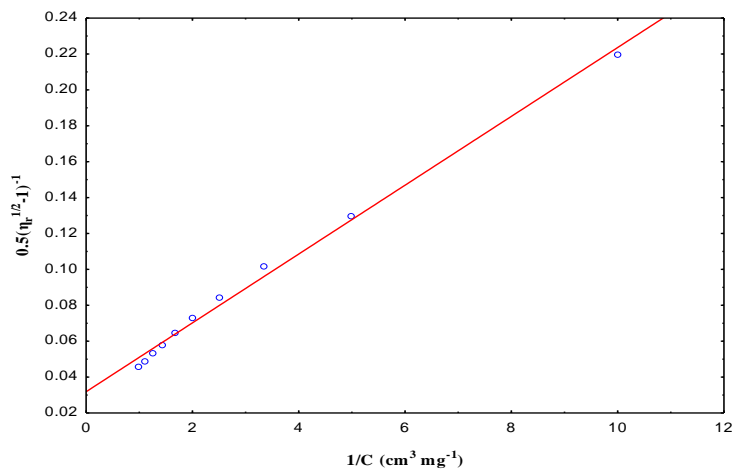


Figure 6 Fedors method

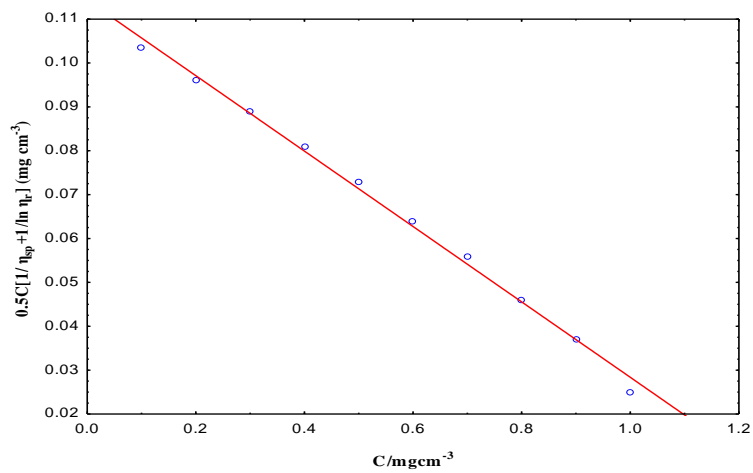




Figure 7 Heller method

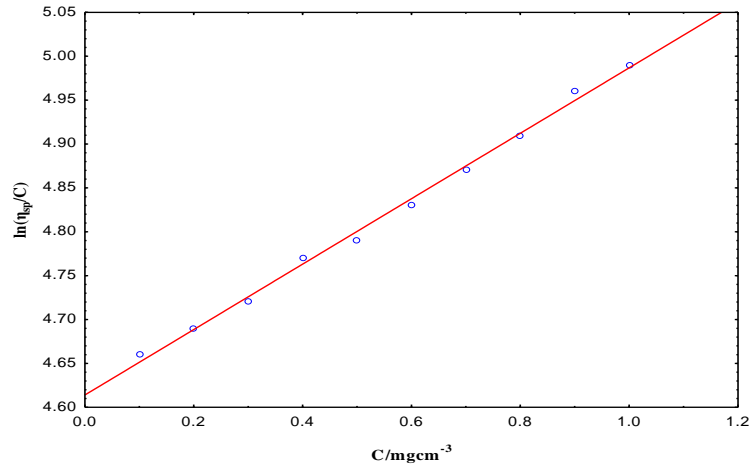


Figure 8 shows Lyons- Tobolsky method

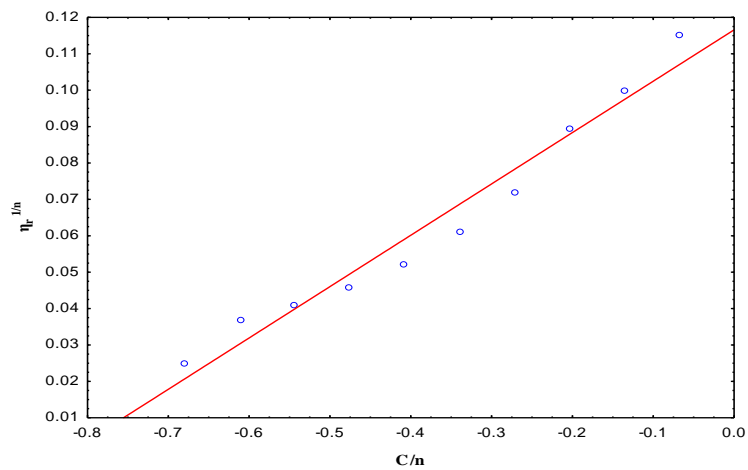


Figure 9 Baker method

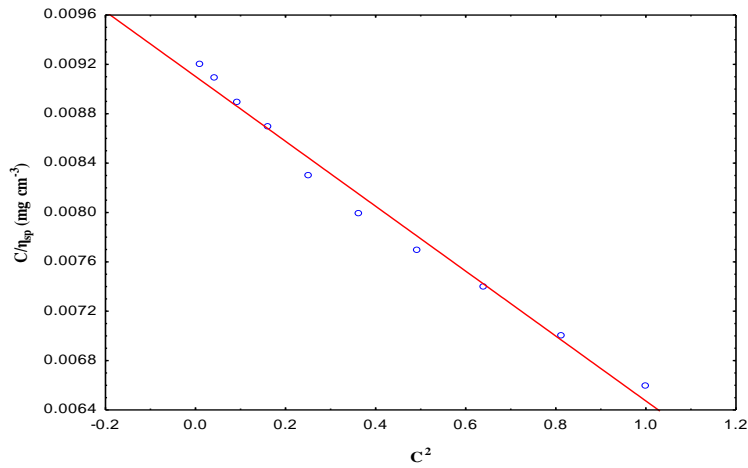


Figure 10 Tager method

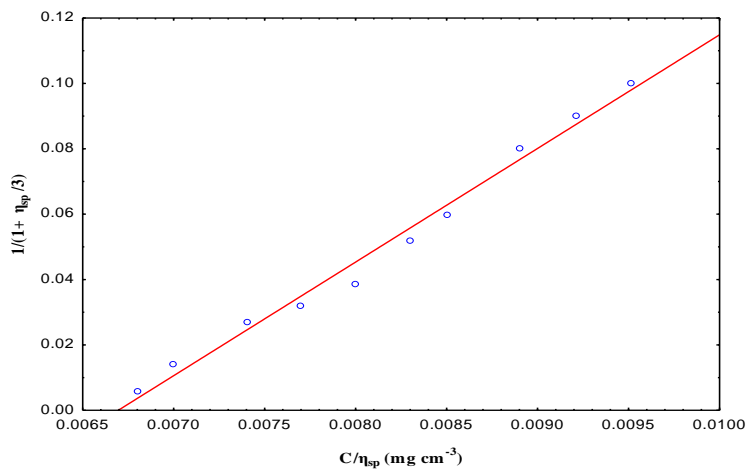


Figure 11 Solomon-Gotesman method

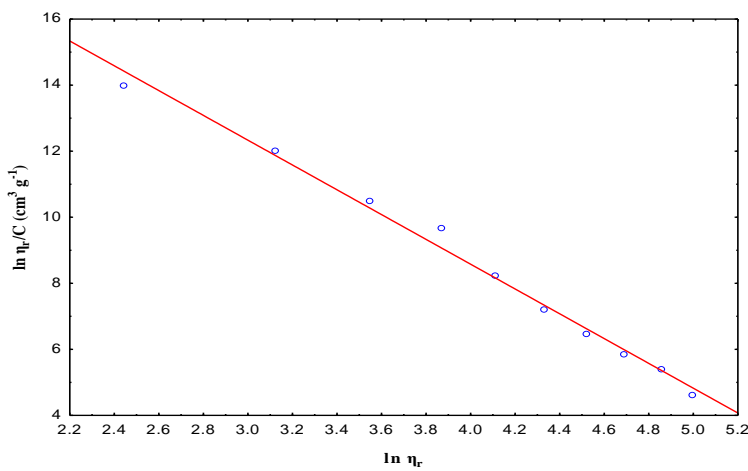


Figure 12 Arrhenius-Rother-Hoffmann method

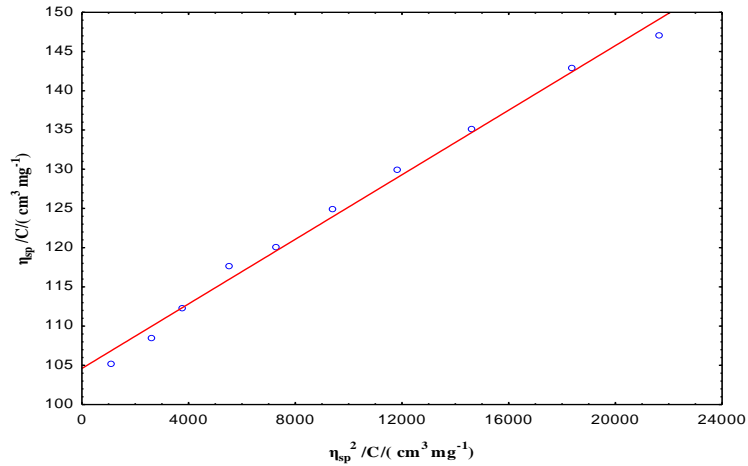


Figure 13 Kreisa method

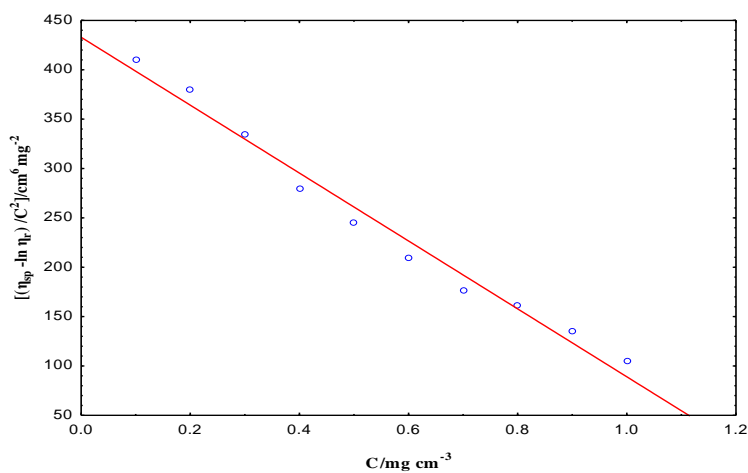


Figure 14 Staudinger-Heuer

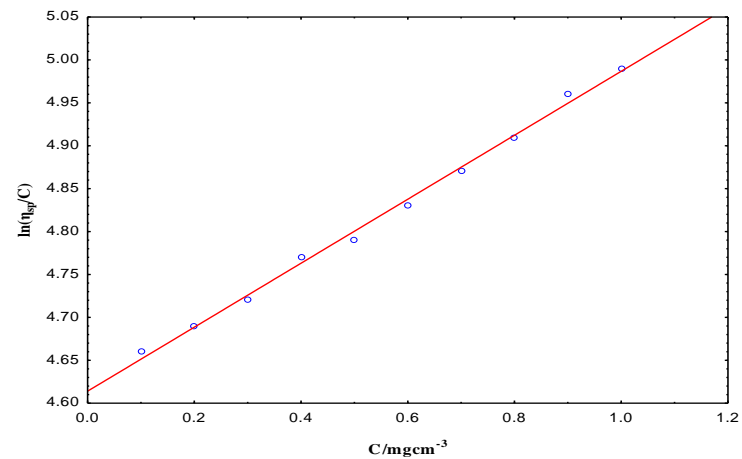




Figure 15 Maron-Reznik method

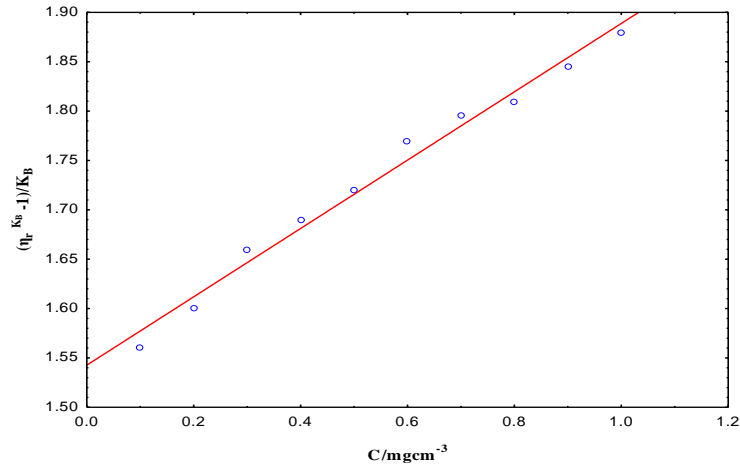


Figure 16 Budtov method

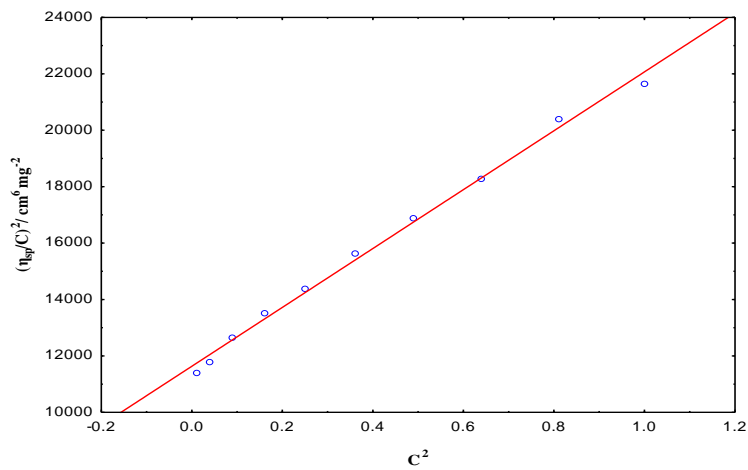


Figure 17 Squire method

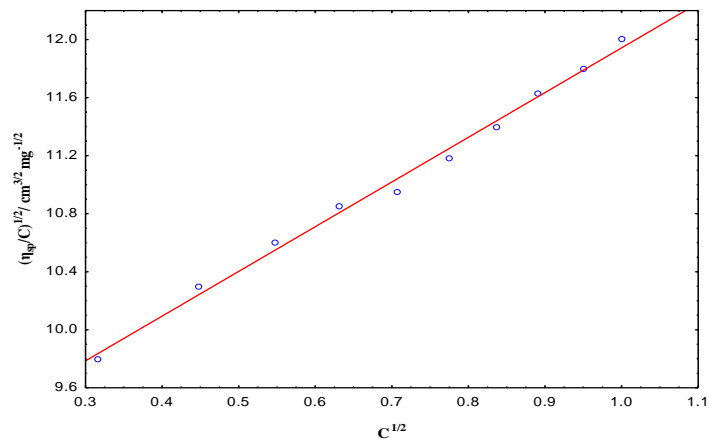




Figure 18 Square root method

In this work, two ways to calculate are proposed that serve to determine the intrinsic viscosity and are plotting methods that take into account the average between two values obtained by different methods that we will detail next. The first plot method has based the equations 23 and 24, plotted in Fig. 17 and Fig. 18. Finally, from the ordinate to the origin, the intrinsic viscosity is obtained and the average is obtained from them.

The relative percentage error concerning Huggins of this equation 23 is by excess (RE%=5.45), and the plot of equation 24 obtained error concerning Huggins is by excess (RE%=2.10). Therefore, an average of both plot solutions gives a value of 1.10% according to the Huggins method.

The mean of intrinsic viscosities values, which were obtained from intercepts in Figure 17 and Figure 18 was $103997.5\text{cm}^3/\text{g}$. The Mark-Houwink parameters observed in Table 2 are referenced by Kim, Choi, Kim, & John [23]. The hydrodynamic radius of the macromolecules changes with this type of solution and with temperature via changes in their chain flexibility. The molecular weight determined for this work is 1043107.58 g/mol , with an intrinsic viscosity by the Huggins method of $98622.4\text{cm}^3/\text{g}$. From clear, this molecule is taken helical conformation shape in solution and the water is a good solvent for it is. Mark-Houwink value of ‘‘a’’ confirms that for these conditions.

Table 2 The Mark-Houwink parameters

K_H	K_M	R_H (nm)	M (g/mol)	$\nu_{a/b}$	$k(\text{cm}^3/\text{g})$	a
0.48	0.57	380	1043107.58	3.30	0.74×10^{-3}	1.35

4. Conclusions

From this research, conclude that the xanthan gum molecule takes the helical conformation in an aqueous solution. According to intrinsic viscosity measurement Folders, Tager, Solomon-Gotesman, and Arrhenius-Rother-Hoffmann's methods are good and so close to Higgins method that is taken as standard. Also, the mean intrinsic value determined in this paper is relevant to the calculation of intrinsic viscosity. Furthermore, it can be concluded that water is an ideal solvent for xanthan gum which is confirmed by the Higgins constant which has a value is about 0.48, and from the large value of intrinsic viscosity, also, the xanthan gum molecule is not related to branching characteristics.

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