

Effect of Sweeteners on Viscosity and Particle Size of Dilute Guar Gum Solutions

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ABSTRACT: *The effects of some synthetic sweeteners on the rheological and physical properties of guar gum in dilute solutions were investigated. Measurements include the determination of intrinsic viscosity and the particle size, surface weighted mean [D3, 2], volume weighted mean [D4,3] and specific surface area of guar gum and synthetic sweeteners mixtures. The concentration of these sweeteners were 0, 0.1, 0.2 % w/w for aspartame, acesulfame-k and cyclamate, and 0, 0.001, 0.002 % w/w for neotame. Gum was evaluated for intrinsic viscosity by various models i.e. Huggins, Kraemer, Tanglertpaibul and Rao equations. The results showed that the values obtained for intrinsic viscosity were different upon various equations used. The plot of relative viscosity versus concentration, obtained from Tanglertpaibul and Rao model described best the phenomenon. Sweeteners had no significant effect on intrinsic viscosity of guar gum solutions.*

KEY WORDS: *Guar gum, Intrinsic viscosity, Sweetener, Rheology.*

INTRODUCTION

Guar gum (GG) is a naturally occurring galactomannan polysaccharide which is made up of a linear chain of b-D-mannopyranose joined by (1-4) linkage with a-D-galactopyranosyl units attached by 1, 6-links in the ratio of 1:2 [1]. It has been suggested as a vehicle for oral controlled release purposes and for colon targeting due to its drug release retarding property and susceptibility to microbial degradation in the large intestine in pharmaceuticals [3-6].

In terms of chemical structure/property relationships, the average galactose content has been shown to strongly influence the physical properties of galactomannans. A lower galactose content yields strong synergistic interactions with other biopolymers and a greater

individual gelling capacity based upon galactose uninhibited mannan interactions [7-9].

The distribution of the galactose units along the main-chain (fine structure) also influences the physical properties of this biopolymer. Whereas it has been shown that the galactose monomers are distributed in a closely random fashion along the mannan backbones galactomannans that possess longer galactose uninhibited mannan regions exhibit greater functionality [10, 11]. In addition, a third and less widely recognized dependence of the chemical structure of galactomannans upon its physical properties, is the influence of the galactose polydispersity between chains. As recently highlighted at the same average galactose content, galactomannans with a broader galactose

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distribution are more functional as they possess a greater proportion of chains with lower galactose content [12].

The intrinsic viscosity, $[\eta]$, is not a very specific parameter and depends upon several factors [13]. It is dependent upon the hydrodynamic volume occupied per unit mass of the macromolecule; which consists of the intrinsic volume occupied by the polymer chain and its excluded free volume. It is also influenced by hydrodynamic properties which include a measure of the permeability of the polymer coil to solvent (if it is free draining, then $[\eta]$ is higher) and chain anisotropy. Deviations from spherical geometry add a frictional component to viscosity. The solution properties of guar gum and other galactomannans in water have been characterized [10].

Rheological property is very important for several aspects such as the effect on mouth feeling, texture, structure and other characteristics of food products. Because gums are widely used for modifying the rheological properties of food systems, investigation on the effect of sweeteners on rheological behavior of gums is very important. By this work, we can conclude that the effect of replacing sucrose with synthetic sweeteners on the rheological property of food products is minimal, and they are suitable alternative sweeteners for sucrose in dietary products.

Goycoolea et al., (1995) removed specific chain/chain association by dissolving guar and locust bean gum neutral polysaccharides in weak polyelectrolyte. This resulted in a significant reduction in the intrinsic viscosity, $[\eta]$, for LBG, 12.1 to 5.2 dl/g and to a lesser extent for guar, 12.5 to 11.9 dl/g. these intrinsic viscosities were then in accordance with the relative actual molecular weights for these two galactomannans. Upon neutralization, the intrinsic viscosities returned to their original values clearly showing that reversible aggregation does occur. Thus, not only does the specific association of galactomannan chains possibly contribute to higher viscosity dependencies with concentration, but it also increases the intrinsic viscosity of the macromolecule (a measure of the hydrodynamic volume) [12]. Sweeteners are a group of widely used low molecular weight additives; however, limited work has studied the influence of sweeteners upon the solution properties of hydrocolloids. For the case of guar and LBG, *Elfak et al.*, (1977) found the intrinsic viscosity to

decrease upon the addition of sucrose and other low molecular weight additives [15]. Recently, in a similar but more extensive study by *Launay et al.* (1997), no change in intrinsic viscosity was measured for 0, 10 and 40 % sucrose concentrations [16].

The aim of this work was to assess the solvent quality of different types of sweeteners and their concentrations for guar gum through measures of the hydrodynamic volume to infinite dilution (through intrinsic viscosity measurements). The objective of this study was to explore the effect of some synthetic sweeteners on the guar gum in dilute solutions.

MATERIALS AND METHODS

Solutions preparation

Guar gum was prepared (C.79, M.I.A. Basni. Phase.II, Joohpur, 342005 (INDIA)). Acesulfame-k and cyclamate were purchased from PT. Golden SARI (Jakarta Utara 14440, Ind Onesia), aspartame and neotame were obtained from nutra sweet company, GC- 02032310.

Sweeteners/galactomannan solutions were prepared by first dry mixing the appropriate amounts of each sweeteners and galactomannan. Cold de-ionised water was then added and the solutions were heated at 60 °C for 15 minutes whilst stirring with a magnetic flea. It was necessary to heat the LBG solutions at such high temperatures to solvate the low galactose, high water temperatures soluble fraction of LBG [12]. Molecular weight analyses showed that the polymer did not degrade at these temperatures unless excessive mechanical stirring was used. Dilute galactomannan solutions were prepared (0.005, 0.01, 0.02, 0.03, 0.04 g/dl) in the ranges of sweeteners concentrations (0, 0.1, 0.2 % w/w for aspartame, acesulfame and cyclamate, and 0, 0.001, 0.002%w/w for neotame) according to *Bayarri et al.*, (2003), [17].

Viscosity measurement

Solution viscosities were determined for a range of shear rate using a rotational viscometer (Brookfield) by spindle No. 2 in the ranges of 0.5-100 rpm at temperature 20 °C. The experimental viscosities, η_{exp} , were converted to relative viscosity, η_{rel} , by subdividing the experimental viscosities to solution viscosity (de-ionised water).

$$\eta_{\text{rel}} = \frac{\eta_{\text{solution}}}{\eta_{\text{solvent}}}$$

Intrinsic viscosity determination

The intrinsic viscosity $[\eta]$ is a measure of the hydrodynamic volume occupied by a macromolecule, which is closely related to the size and conformation of the macromolecular chains in a particular solvent [18, 19]. The intrinsic viscosity $[\eta]$ was measured according to the *Higiro et al.* (2006). According to their method the intrinsic viscosity $[\eta]$ is determined experimentally from measurements of the viscosity of very-low-concentration (C) solutions. Denoting solution and solvent viscosity as, respectively, η_{solution} and η_{solvent} , $[\eta]$ is defined by the following relationships:

$$\text{Relative viscosity: } \eta_{\text{rel}} = \eta_{\text{solution}} / \eta_{\text{solvent}} \quad (1)$$

$$\text{Specific viscosity: } \eta_{\text{sp}} = \eta_{\text{rel}} - 1 \quad (2)$$

$$\text{Intrinsic viscosity: } [\eta] = \lim_{c \rightarrow 0} \frac{\eta_{\text{sp}}}{c} \quad (3)$$

The intrinsic viscosity can be obtained by measuring specific viscosities at different concentrations at the same shear-rate, and extrapolating the course of specific viscosity to infinite dilution [19, 20]. The intrinsic viscosity $[\eta]$ is, therefore, obtained by extrapolating data to zero concentration by using a linear regression, which will be called the graphic double-extrapolation procedure (GDEP) in this study. *McMillan* (1974) showed that η_{sp}/c , also called reduced viscosity, could be written in the form of a *Huggins* equation [21, 22],

$$\frac{\eta_{\text{sp}}}{c} = [\eta] + k'[\eta]^2 c \quad (4)$$

where k' is the *Huggins* constant. The determination of the intrinsic viscosity is, therefore, the extrapolation of reduced viscosity to the value at zero solute concentration.

The extrapolations are usually done in very dilute regimes ($C \ll C^*$) with relative viscosity values between 1.2 and 2.0, the corresponding specific viscosities being between 0.2 and 1.0 [23]. C^* is defined as the overlap concentration, the transition from the dilute to the semi-dilute region which marks the onset of polymer entanglement [16]. In addition, *McMillan* (1974) reported that the intrinsic viscosity could be obtained from the *Kraemer* equation [21, 24] by extrapolation to zero concentration (C).

$$\frac{\ln \eta_{\text{rel}}}{C} = [\eta] + k''[\eta]^2 \quad (5)$$

Where k'' is the *Kraemer* constant. For very dilute solutions, however, Eq. (5) can be shortened by retaining only the first-order term, and $[\eta]$ can be determined from the slope of a plot of C against $\ln \eta_{\text{rel}}$ [25]. *McMillan* (1974) showed that methods of determination of the intrinsic viscosity that were based on slopes of plots had higher correlation coefficients and lower standard errors, compared with those based on intercepts of plots [21]. On the basis of such findings, *Tanglertpaibul* and *Rao* (1987) used the following equations to obtain the intrinsic viscosity of tomato serum [26]:

$$\eta_{\text{rel}} = 1 + [\eta]C \quad (6)$$

The intrinsic viscosity $[\eta]$ is the slope obtained by plotting η_{rel} vs. C

$$\eta_{\text{rel}} = e^{[\eta]C} \quad (7)$$

The intrinsic viscosity $[\eta]$ is the slope obtained by plotting $\ln \eta_{\text{rel}}$ vs. C

$$\eta_{\text{rel}} = \frac{1}{1 - [\eta]c} \quad (8)$$

The intrinsic viscosity is the slope obtained by plotting $1 - \frac{1}{\eta_{\text{rel}}}$ vs. C

The intrinsic viscosity $[\eta]$ was estimated based on the slope of η_{sp} vs. C for polyelectrolytes, as suggested by *Chou* and *Kokini* (1987); this is similar to the method discussed in Eq. (6). *Chou* and *Kokini* (1987) reported that when there is essentially no molecular interaction, as in dilute solutions, the second term of the *Huggins* equation (Eq.(4)) is negligible, and a plot of η_{sp} against concentration is linear. In this study, the intrinsic viscosity in the dilute domain was estimated on the basis of Eqs. (3), (6), (7), and (8), and the four methods were statistically compared for a better fit [27].

Particle size distribution

Particle size distribution was determined by integrated light scattering using a Mastersizer 2000 (Malvern Instruments Ltd., Malvern, UK). The emulsions were analyzed immediately after preparation in duplicate. The instrument used the principle of *Fraunhofer* diffraction where a parallel, monochromatic beam of laser light (red light = 633 nm) illuminates the suspensions. The light diffracted by the suspension droplets gives a stationary

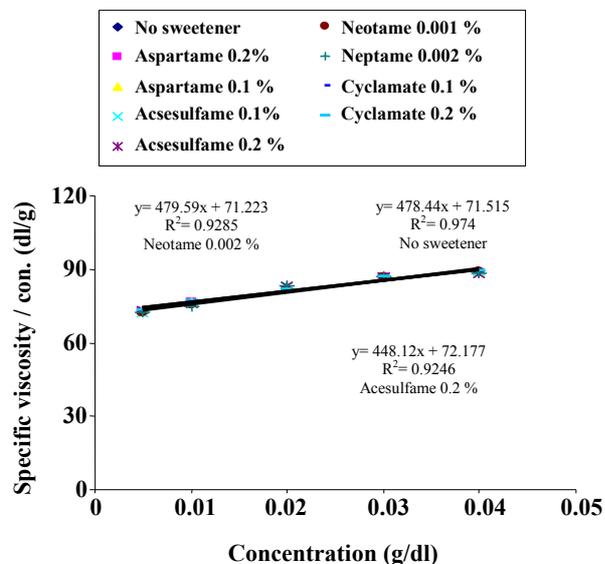


Fig. 1: Huggins plot (specific viscosity/concentration vs. concentration) for guar gum with addition of sweeteners.

diffraction pattern regardless of the particle movement. As particles enter and leave the illuminated area, the diffraction pattern changes, always reflecting the instantaneous size distribution in the illuminated area⁸. The particle diameter range, specific surface measurements were performed at room temperature (20 °C). A 45 mm focal lens was used for the measurements. The average droplet size was characterized by the mean diameter d_{43} and d_{32} defined by:

$$d_{43} = \frac{\sum_i n_i d_i^4}{\sum_i n_i d_i^3} \quad (9)$$

and

$$d_{32} = \frac{\sum_i n_i d_i^3}{\sum_i n_i d_i^2} \quad (10)$$

Where n_i is the number of droplets of diameter d_i . The d_{43} and d_{32} values were used to monitor changes in the droplet-size distribution of freshly made solutions with different compositions.

Statistical analysis

A two-way factorial design was used to generate the best-fitting intrinsic viscosity model. For each gum, the three sweeteners (aspartame, acesulfame, cyclamate and neotame) at three concentrations (0, 0.1, and 0.2 % w/w for aspartame, acesulfame and cyclamate, and 0, 0.001, 0.002 % w/w for neotame) were compared for the intrinsic

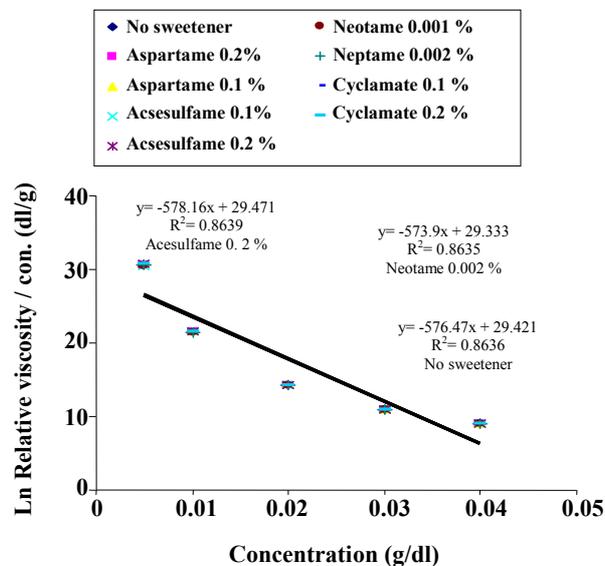


Fig. 2: Kraemer plot (Ln relative viscosity/concentration vs. concentration) for guar gum with addition of sweeteners.

viscosity, in a factorial design. In each instance, three replications were made. The analysis of variance and means comparison were conducted by the general linear models procedure (Proc GLM), with statistical analysis system software (version 9.1, SAS Institute, Inc., Cary, NC). Comparisons among treatments were analyzed by using Fisher's least significant difference (LSD), with a significance level at $P < 0.05$.

RESULTS AND DISCUSSION

Intrinsic viscosity

In general, for Guar gum, an increase in specific viscosity was observed as the concentration of gum increased. The same results have been reported by Lapasin and Pricl (1995), indicating that neutral polysaccharides (i.e., guar) exhibited linear plots of lower slope, whereas ionic polysaccharides displayed a sharp increase of the slope, possibly due to expanded coil dimensions and electrostatic repulsion between chain segments [28].

We observed the same results for guar gum (Fig. 1). Fig. 1 displays increasing of specific viscosity/ concentration as concentration increase.

Lai et al. (2000) reported similar results when determining the intrinsic viscosity of hsian-tso leaf gum in different salt solutions [18]. Other works showed the same results for salts [19, 29].

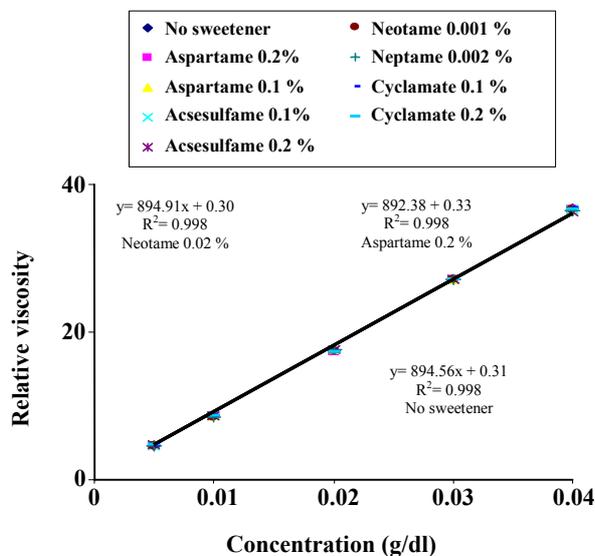


Fig. 3: Relative viscosity for guar gum as a function of gum concentration with addition of sweeteners.

The data from *Huggins* plot showed that at applied sweetener concentrations there was no significant effect on IV (Fig. 2). The non-linear relationship between $\ln \eta_{rel} / C$ and gum concentration (*Kraemer* model) was observed, thus making impossible the determination of the intrinsic viscosity by extrapolation of experimental data with this equation; this prompted the use of slope models and *Huggins* plot [24, 26, 27].

From Eq. (4), the *Huggins* constant k' theoretically should lie between 0.3 and 0.8, and values larger than 1 represents polymer-polymer aggregation, increasing as the solvent quality decreases, resulting in polymer coil contraction and larger values possibly indicate a poorer solvent [13, 30]. It was found that the k' values lied in the range of 0.03- 0.09, meaning molecular association was absent and solvent quality was good. This was in accordance with the sum of the *Huggins* constant k' and *Kraemer* constant k'' which equals $0.5 \pm 10\%$, and larger or smaller values are attributed to molecule association [19, 31].

To determine the intrinsic viscosity by plotting relative viscosity vs. C (Eq. (6)) and $\ln \eta_{rel}$ vs. C (Eq. (7)), straight-line relationship with large linear regression coefficients were obtained (Figs. 4, 5). In the case of $1/\eta_{rel}$ vs. C (Eq. (8)) straight-line relationship with low linear regression coefficients was obtained (Fig. 6). *McMillan* (1974) reported that methods of determination of intrinsic viscosity based on slopes of plots had larger

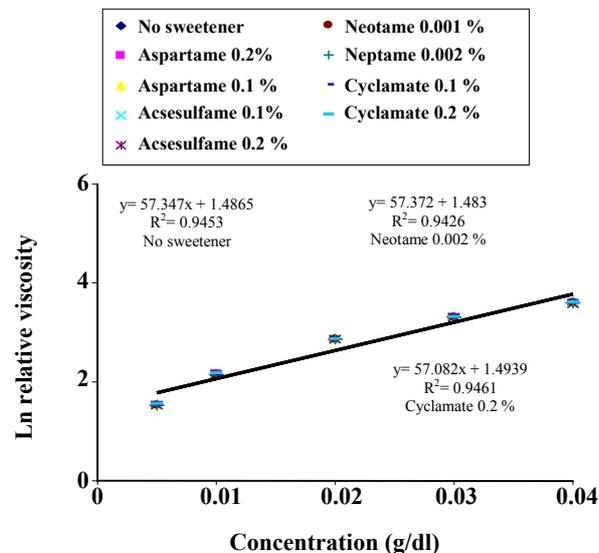


Fig. 4: Ln Relative viscosity for guar gum as a function of gum concentration with addition of sweeteners.

correlation coefficients and smaller standard errors than did those based on intercepts of plots [21]. The representative comparison of the different models used to estimate the intrinsic viscosity is shown for each gum (table 1).

Intrinsic viscosity values calculated by using Eq. (6) were larger and significantly different from those obtained using Eqs. (7) and (8) for all sweeteners and sweetener concentrations (Fig. 3). *Tanglertpaibul* and *Rao* (1987) successfully implemented the model from Eq. (6) in the intrinsic viscosity determination of tomato serum [26]. The authors reported higher correlation coefficients and fewer errors. Significant differences between guar gum were detected with Eq. (6), whereas these differences were few with Eqs. (7) and (8). We used Eq. (6) as the best del for intrinsic viscosity determination because it showed a better linear fit, with higher correlation (R^2) for gums, sweeteners, and sweetener concentrations. Furthermore, the results were similar to other findings [26]. *Richardson et al.* studied the effect of sucrose on dilution solution properties of guar gum. Their results showed that addition of sucrose at low concentrations (0-10 % w/w) decrease the intrinsic viscosity (because of decreasing the solvent quality), and these results could compliment the *Huggins* model. Sucrose influences the intrinsic viscosity by changing structure of the hydrocolloid (coil expansion) and so the rheological behavior.

Our results showed that the values obtained for intrinsic viscosity were different by the equation used for

Table 1: Intrinsic viscosity values (dl/g) obtained for guar gum.

Sweetener	Sweetener con. (%)	1	2	3	4	5
Aspartame	0	71.51±2.9 _a ^B	29.42±1.7 _a ^D	894.56±4.7 _a ^A	57.35±2.3 _a ^C	4.79±0.3 _a ^E
	0.1	72.30±2.6 _a ^B	29.52±1.2 _a ^D	894.34±4.2 _a ^A	57.16±2.5 _a ^C	4.76±0.4 _a ^E
	0.2	71.56±2.3 _a ^B	29.42±1.6 _a ^D	892.38±5.6 _a ^A	57.25±2.1 _a ^C	4.79±0.3 _a ^E
Acesulfame k	0	71.51±2.9 _a ^B	29.42±1.7 _a ^D	894.56±4.7 _a ^A	57.35±2.3 _a ^C	4.79±0.3 _a ^E
	0.1	71.22±2.7 _a ^B	29.31±1.1 _a ^D	892.43±6.0 _a ^A	57.52±2.2 _a ^C	4.83±0.2 _a ^E
	0.2	72.18±3.0 _a ^B	29.99±1.1 _a ^D	891.18±5.6 _a ^A	57.02±2.4 _a ^C	4.77±0.4 _a ^E
Cyclamate	0	71.51±2.9 _a ^B	29.42±1.7 _a ^D	894.56±4.7 _a ^A	57.35±2.3 _a ^C	4.79±0.3 _a ^E
	0.1	71.11±3.1 _a ^B	29.62±1.4 _a ^D	894.46±5.1 _a ^A	57.44±2.6 _a ^C	4.82±0.2 _a ^E
	0.2	72.19±2.8 _a ^B	29.83±1.1 _a ^D	894.91±6.2 _a ^A	57.08±2.8 _a ^C	4.76±0.2 _a ^E
Neotame	0	71.51±2.9 _a ^B	29.42±1.7 _a ^D	894.56±4.7 _a ^A	57.35±2.3 _a ^C	4.79±0.3 _a ^E
	0.001	70.39±1.9 _a ^B	29.23±1.2 _a ^D	894.15±5.9 _a ^A	57.69±2.1 _a ^C	4.85±0.2 _a ^E
	0.002	71.22±2.1 _a ^B	29.33±1.3 _a ^D	892.05±5.3 _a ^A	57.37±2.3 _a ^C	4.82±0.3 _a ^E

Results are expressed as means ± SD for three replications.

A-E: Means followed by the same letters in the same row are not significantly different ($P < 0.05$).

a-b: Means followed by the same letters in the same column are not significantly different ($P < 0.05$).

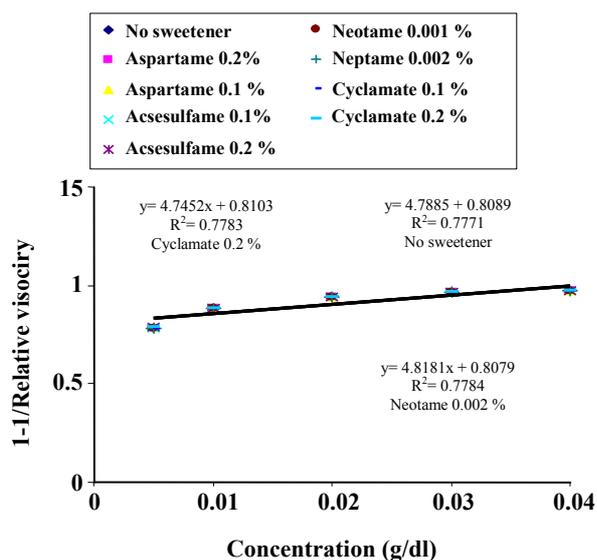


Fig. 5: Plot of $(1-1/\text{Relative viscosity})$ as a function of gum concentration for guar gum with addition of sweeteners.

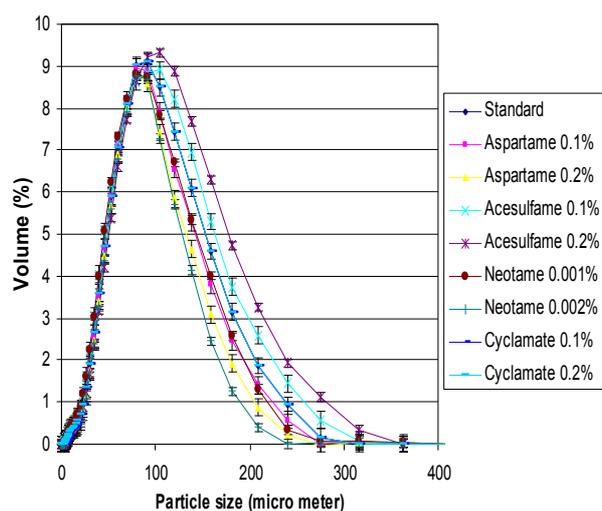


Fig. 6: Relative viscosity for guar gum as a function of gum concentration with addition of sweeteners.

Table 2: The particle size distribution (d0.1, d0.5 and d0.9), surface weighted mean, volume weighted mean, specific surface area for guar gum suspension at 0.5 % concentration.

Sweetener	Particles size distribution			Surface weighted	Vol. weighted	Specific surface
	d0.1	d0.5	d0.9	Mean [D3,2] μm	Mean [D3,4] μm	Area ($\mu\text{m}^2/\text{g}$)
No sweetener	40.33	90.02	184.24	70.17	103.77	0.085
Aspartame 0.1 %	39.12	88.77	181.64	63.96	102.41	0.088
Aspartame 0.2 %	37.94	90.07	177.09	60.45	99.55	0.108
Acesulfame 0.1 %	42.73	91.01	186.62	69.14	105.85	0.089
Acesulfame 0.2 %	40.22	93.31	191.94	77.83	109.36	0.080
Neotame 0.001 %	38.25	91.14	1858.21	64.66	97.82	0.108
Neotame 0.002 %	36.17	89.30	178.47	59.49	98.19	0.105
Cyclamate 0.1 %	40.60	90.16	184.16	67.20	103.15	0.097
Cyclamate 0.2 %	39.21	92.70	183.99	67.73	104.86	0.091

estimation the intrinsic viscosity. We found *Kraemer* equation and $1-1/\eta_{\text{rel}}$ versus concentration plot did not provide a better fit for guar gum. Addition of these sweeteners in these ranges of concentration had no significant effect on intrinsic viscosity of guar gum solutions, indicating that these synthetic sweeteners had no effects on solvent quality and hydrodynamic volume of guar gum.

$$\frac{\eta_{\text{sp}}}{c} = [\eta] + k'[\eta]^2 c \quad (11)$$

$$\frac{\ln \eta_{\text{rel}}}{c} = [\eta] + k''[\eta]^2 c \quad (12)$$

$$\eta_{\text{rel}} = 1 + [\eta]c \quad (13)$$

$$\eta_{\text{rel}} = e^{[\eta]c} \quad (14)$$

$$\eta_{\text{rel}} = \frac{1}{1 - [\eta]c} \quad (15)$$

Particle size distribution

The particle size distribution of the suspension droplets was determined on different sugar suspensions. As it can be seen from Fig. 6 and table 2, the addition of sweeteners on the gum solutions causes a brief change in the particle size. The effects of sweetener and its concentration on the particle size distribution are shown

in table 2. There is a difference between acesulfame k and other sweeteners behavior, as increasing the concentration of acesulfame k causes an increase in particle size, surface weighted mean [D3,2], volume weighted mean [D4,3], and decrease in specific surface area. However, the particle size, surface weighted mean [D3, 2], volume weighted mean [D4,3], of the suspension made by aspartame and neotame decreased which the specific surface area increased. In the case of cyclamate, the changes of these parameters were insignificant. These different effects among various sweeteners may be due to their different molecular structures. Smaller particle size can lead to more stable suspension, indicating that the solutions formulated with acesulfame k may be unstable than those of other synthetic sweeteners.

CONCLUSIONS

The *Kraemer* and $1/1-\eta$ relative plots do not provide a better fit for guar gum with addition of aspartame, acesulfame, cyclamate and neotame in regular concentrations. To determine the intrinsic viscosity by plotting specific viscosity/concentration versus C (Huggins equation, (Eq.(4)), relative viscosity vs. C (Eq. (6)), $\ln \eta_{\text{rel}}$ vs. C (Eq. (7)), straight-line relationships with large linear regression coefficients were obtained for guar gum but among of them we found relative viscosity versus gum concentration plot (Eq.(6)), as the best model for its

higher linear regression coefficient. Using any of equations results in difference values for intrinsic viscosity. By using these appropriate equations, there are not any significant different among these sweeteners, indicating these synthetic sweeteners had no effects on solvent quality and the hydrodynamic volume of guar gum may be due to their low concentrations. It is possible that when their concentrations increase, their effects become significant.

The effects of these synthetic sweeteners are different. There is a difference between acesulfame k and other sweeteners behavior, as acesulfame k causes an increase in particle size, surface weighted mean [D3,2], volume weighted mean [D4,3], and decrease in specific surface area. However, the particle size, [D3, 2], [D4, 3], of suspension made by aspartame and neotame decreased and specific surface area increased. In the case of cyclamate, the changes of these parameters of suspension were insignificant. The results suggested that the solutions formulated with acesulfame k may be unstable than that of other synthetic sweeteners.

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