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TEMPERATURE AND SALINITY INFLUENCE ON RHEOLOGY OF AQUEOUS DIUTAN GUM SOLUTION

Roberto Guimarães Pereira[1], Guilherme Pereira Mota[2], Ithamar Ribeiro Rangel[3] and João Crisósthomo de Queiroz Neto[4]
[1]Fluminense Federal University - UFF/TEM/PGMEC/PGEB/MSG, Rua Passo da Pátria, São Domingos - CEP: Niterói, RJ, Brazil
[2]Fluminense Federal University - UFF/PGMEC, Rua Passo da Pátria, São Domingos - CEP, Niterói, RJ, Brazil
[3]Geraldo Di BiaseUniversity Centre, Rua Dep.Geraldo Di Biase, Volta Redonda-RJ, Brazil
[4]Fluminense Federal University - UFF/ Petroleum Engineering Department, Rua Passo da Pátria, São Domingos - CEP, Niterói, RJ, Brazil
E-Mail: temrobe@vm.uff.b

ABSTRACT

The temperature and salinity influence on rheology of aqueous diutan gum solution at the temperatures of 20°C, 40°C and 60°C were investigated. It was used a solution of diutan gum (4300 ppm) in deionized water and a solution of diutan gum (4300 ppm) in deionized water with 40000 ppm of NaCl. Steady shear, creep-recovery and oscillation tests were performed. Aqueous diutan gum solutions showed a pseudoplastic and viscoelastic behavior. The viscosity of aqueous diutan gum solution changes very litle in the presence of NaCl. With the salt addition, at 60^0C, the viscous modulus keep higher than the elastic modulus in the frequency range investigated. In the others cases, for angular velocities greater than an specific value, the elastic modulus keep higher than viscous modulus indicating a gel behavior of diutan aqueous solutions. The Cox-Merz rule is better applicable in the case of diutan gum solution (4300 ppm) at higher temperature.

Keywords: biopolymer; viscosity; viscoelasticity; power-Law model; exponecial model.

1. INTRODUCTION

Microbial polysaccharides (xanthan gum, welan gum, guar gum, diutan gum) are used in many areas as pharmaceutics, cosmetics, foods, oil recovery, soil strengthening, cementation and flow controlling fluids for thickening, emulsifying, stabilizing or gelation (Xu *et al*., 2015; Chang *et al*., 2015; Lee *et al*., 2015; Lee and Song, 2015; Xu *et al*., 2014; Khalil and Jan, 2012; Sonebi and McKendry, 2008; Zsivanovitis *et al*., 2007; Dolz*et al*., 2006; Gebert and Friend, 1998). They are biocompatible, biodegradable, and environmentally Friendly (Xu *et al*., 2015; Chang *et al*., 2015; Khalil and Jan, 2012).

Diutan gum, a novel microbial polysaccharides, is an anionic biopolymer and consists of a repeat unit with β-1, 3-D-glucopyranosyl, β-1, 4-D-glucuronopyranosyl, β-1,4-D-glucopyranosyl, and α-1, 4-L-rhamnopyranosyl, and a twosaccharideL-rhamnopyranosyl side-chain attached to the (1→4) linked glucopyranosyl residue(Xu *et al*., 2015; Banerjee *et al*., 2009). Diutan gum is a natural high molecular weight microbial polysaccharide; secreted by the bacterium Sphingomonas sp. Diutan gum has a regular double helix molecular conformation in aqueous solution.
Diutan gum has many applications: it is an effective additive for improving the viscosity and the performance of cement paste (Zhang *et al*., 2010); it has a great potential in tertiary oil recovery because of its distinctive physicochemical properties under harsh reservoir conditions, such as high temperature and high salinity (Li *et al*., 2017). Diutan gum acts as: stabilizing; thickening, binding; emulsifying and suspending agent (Xu *et al*., 2015).

Few works about diutan gum have been reported in the literature. Some of these are as follows.

Li *et al.* (2017) evaluated the potential of diutan gum, for enhanced heavy oil recovery at high temperature and high salinity. They found that the steady apparent viscosity and dynamic modulus of aqueous diutan gum solutions are not sensitive to the temperature and virtually independent of the salinity. They pointed out that the gel like structure of diutan gum is dependent on the shear rate rather than the shear time and the aging time. They realized sandpack flooding experiments showing that the heavy oil recovery efficiency of diutan gum is raised by 20.9%. They concluded that diutan gum will be a promising oil recovery agent for enhanced oil recovery in high-temperature and high-salinity reservoirs.

Xu *et al*. (2015) studied the rheological properties of diutan gum in aqueous solution. They showed that the molecular aggregates of diutan gum can be formed at a very low concentration (0.12 $g.L^{-1}$). The gel structure can be formed in the diutan gum solution and the gelproperties are not sensitive to temperature, and are virtually independent of cationic environment (Na^+ and Ca^{2+}). They conclude that the temperature/salt tolerance of the diutan gum solution is mainly attributed to its perfect double helix molecular conformation, the location of the side chains of its molecules, and its water retention capacity.

Zhang *at al*. (2010) studied the early hydration and setting of cement pastes. The diutan gum was used as additive. They conclude that diutan gum delay cement hydration, causing later setting times.

Barnerjee *et al*. (2009) studied different gums including diutan gum. Properties such as water vapor adsorption, their hydration in solution, their viscosity behaviors, and salt effects on fluidity were studied. The viscosity values of the gums have followed the power law equation,

Sonebi (2006) evaluated the influence of the dosage of the second generation of viscosity modifying agent (diutan gum) on fluidity and rheological parameters of cement-based materials grout compared to welan gum. It was shown that for a given dosage of viscosity modifying agent, diutan gum showed a high apparent viscosity than welangum which could be attributed to the molecular weight and to the long-side chain of diutan gum leading to greater entanglement and intertwining.

In this paper, we investigated the temperature and salinity influence on rheology of aqueous diutan gum solution at the temperatures of 20°C, 40°C and 60°C. It was used a solution of diutan gum at the concentration of 4300 ppm in deionized water and a solution of diutan gum at the concentration of 4300 ppm in deionized water with 40000 ppm of NaCl. The use of NaCl in the solutions is justified because it is the salt of the highest percentage present in sea water and reservoirs, and the evaluation of the influence of the temperature becomes necessary since it varies according to the oil well to be explored. Steady shear, creep-recovery and oscillation tests were performed, yielding the following rheological properties, among others: viscosity (η); elastic modulus (G'); viscous modulus (G''); complex modulus (G^*) and complex viscosity (η^*). A Power Law model it was used to fit the relationship between viscosity and shear rate. An exponential model and a Power Law model were used to describe the behavior of G' and G'' as a function of angular velocity (w). The Cox-Merz rule is better applicable in the case of diutan gum solution (4300 ppm) at higher temperature (60^0C). Aqueous diutan gum solution showed a pseudoplastic and viscoelastic behavior, even with salt addition.

2. MATERIALS AND METHODS

2.1 Sample Preparation

Two different solutions of diutan gum manufactured by CP Kelco were used in the tests: diutan gum (4300 ppm) added as a powder in deionized water and diutan gum (4300 ppm) added as a powder in deionized water with 40000 ppm of NaCl. The solutions stayed under mechanical shaking at room temperature of 25 ° C, with the angular velocity of the mixer at 500 rpm for 24 hours and after that, they were left resting for 24 hours and, then, submitted to the deformation tests.

2.2 Determination of Rheological Properties

The tests were performed on a Haake RS50 rotational rheometer using cone-plate geometry (C35/2° Ti sensor, with a diameter of 35 millimeters and 2° of conicity). Steady shear, creep-recovery and oscillation tests were performed at temperatures of 20°C; 40°C and 60°C, yielding the following rheological properties, among others: viscosity; elastic modulus; viscous modulus; complex modulus and complex viscosity. The maximum allowable temperature deviation was±0.2^0C. Samples were kept for more than 24h before measurements to guarantee the absence of bubbles.

2.2.1 Steady shear tests

The rheological parameters were determined by the controlled stress (CS) test, in the range of 0.001 to 10000 s^{-1}.

2.2.2 Creep and recovery tests

In the creep stage, a constant shear stress of 1 Pa was applied to the sample for 120s, then the shear stress was set to zero for 120s during the recovery. The deformation were measured of a function of time

2.2.3 Oscillation tests

The linear viscoelastic region was determined by the amplitude stress sweep test, keeping the angular frequency in 1 Hz.

In the frequency sweep tests, a stress of 1Pa, determined through the amplitude stress sweep test, was selected to ensure that the samples were within the linear viscoelastic region.

2.2.4 Structural recovery tests

The structural recovery of the samples was determined according to the procedure of Mezger (2006) with some modifications. The samples were deformed by shearing at temperatures of 20°Cand 60°C following these steps: (1) controlled strain rate of 5 $s^{-1}$was applied for 120s and thereafter (2) controlled strain rate of 15$s^{-1}$was applied for 60 s and (3) controlled strain rate of 5 $s^{-1}$was applied for 180s. The apparent viscosity recovery percentage was calculated by the ratio of the average viscosity obtained in the step 3 and of the step 1.

3. RESULTS AND DISCUSSIONS

3.1 Steady shear tests

The Figure 1A shows the influence of shear rate on the viscosity of ditan gum solution (4300 ppm) at 20^0C, 40^0C and 60^0C. The diutan gum solution shows apseudoplastic behavior. Higher the temperature, greater are the reduction in viscosity for all shear rate investigated.

The influence of shear rate on the viscosity of ditan gum solution (4300 ppm) with NaCl (40000 ppm) at 20^0C, 40^0C and 60^0C is shown in Figure1B. The diutan gum solution with NaCl (40000ppm) also shows a pseudoplastic behavior. At higher temperature, the reduction in viscosity for all shear rate investigated is not accentuated because of the presence of NaCl on diutan gum solution.

The pseudoplastic behavior of diutan gum solution with and without NaCl is also reported at the literature (Xu *et al*., 2015; Li *et al*., 2017).

As shown in Figure-1C at 20^0C and Figure-1D at 60^0C the viscosity of diutan gum solution and of diutan gum solution with NaCl are very close. At 60^0C (Figure1D) the viscosity of diutan gum solution with NaCl are littlehigher than of diutan gum solution. Xu *et al.* (2015) also reported that the apparent viscosity of aqueous diutan gum solution changes negligibly in the presence of inorganic salts.

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Different fluids, at low shear rate, exhibits a constant value of viscosity (plateau) indicating in this range of shear rate a Newtonian behavior. In the present study, this trend was more pronounced in the case of aqueous diutan gum solution (4300 ppm) without NaCl for shear rate less than $0.04s^{-1}$ (Figure-1A).

Table-1 shows the parameters of the Power Law Model (Equation 1) for the diutan gum solution and of diutan gum solution with NaCl (Figure-1A and Figure-1B).

$$\eta = K\dot{\gamma}^{n-1} \quad (1)$$

where k is the consistency index and n is the power-law exponent (flow behavior index)

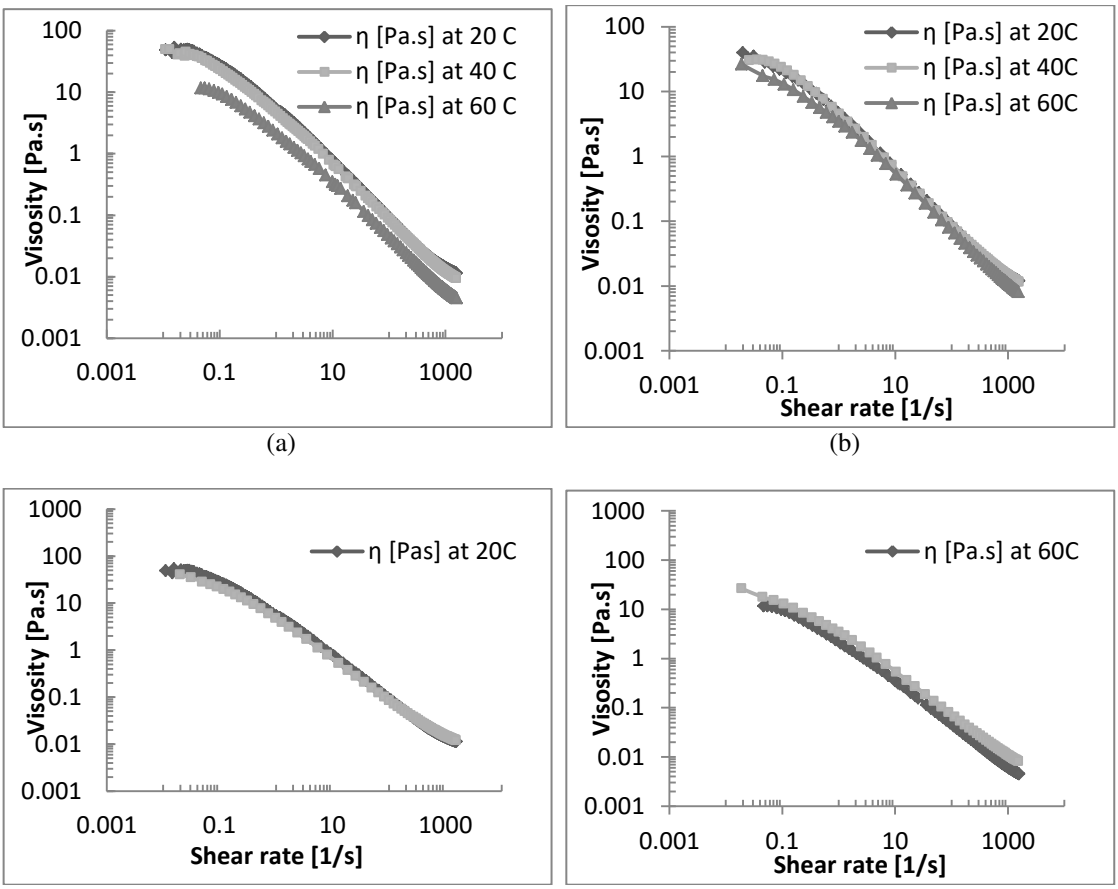


Figure-1. (A) Flow curve at 20^0C, 40^0C and 60^0C of diutan gum solution (4300ppm); (B) Flow curve at 20^0C, 40^0C and 60^0C of diutan gum solution (4300ppm) with NaCl (40000 ppm); (C) Flow curve at 20^0C of diutan gum solution (4300ppm) and diutan gum solution (4300ppm) with NaCl (40000 ppm); (D) Flow curve at 60^0C of diutan gum solution (4300ppm) and diutan gum solution (4300ppm) with NaCl (40000 ppm)

Table-1. Parameters of the Power Law Model for the diutan gum solution and of diutan gum solution with NaCl.

			TEMPERATURE (^{0}C)	
		20	**40**	**60**
DIUTAN GUM	K [Pa.s^n]	7.390	6.705	2.558
	n	0.467	0.539	0.468
	R^2	0.9886	0.9602	0.9882
DIUTAN GUM With NaCl	k [Pa.s^n]	6.023	6.593	3.831
	n	0.497	0.523	0.503
	R^2	0.9904	09704	0.9922

R - Correlation Coefficient

Other authors have also reported the use of Power Law model to fit the relationship between viscosity and shear rate in the case of aqueous solution of microbial polysaccharides (Xu *et al*., 2013) in the case of welan gum and xanthan gum; Marcotte *et al*., (2001) in the case of xanthan gum and Wang *et al.* (2009) in the case of xanthan gum).

3.2 Creep and recovery tests

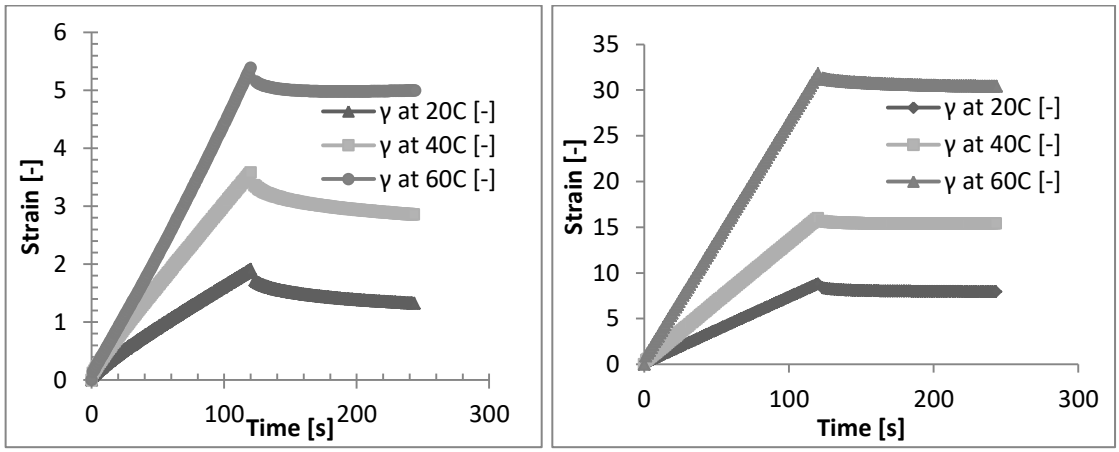
As shown in Figure-2A and Figure-2B, the recovery is more accentuated in the case of diutan gum without NaCl. At higher temperature, the deformation (strain) is greater. This recovery of diutan gum with and without NaCl is an indicator of the sample viscoelastic behavior. Table-2 shows the recovery rate of diutan gum with and without NaCl for the temperatures of 20^0C, 40^0C and 60^0C.

The recovery rate was used to identify the recovery percentage of the sample, and was defined as:

$$\text{Recovery rate [\%]} = [(\gamma_T - \gamma_P)/\gamma_T] \times 100 \quad (2)$$

where: γ_T is the total strain and γ_P is the permanent strain

The recovery rate (Equation 2) has also been used by Wu *et al*. (2010) to identify the recovery percentage of extruded flaxseed-maize pastes.



(c) (d)

Figure-2. (A) Creep and recovery at 20^0C, 40^0C and 60^0C of diutan gum solution (4300ppm); (B) Creep and recovery at 20^0C, 40^0C and 60^0C of diutan gum solution (4300ppm) with NaCl (40000 ppm)

Table-2. Percentage of recovery of diutan gum solution with and without NaCl for the temperatures of 20^0C, 40^0C and 60^0C.

			TEMPERATURE (^{0}C)	
		20	**40**	**60**
DIUTAN GUM	Recovery rate [%]	30.6	20.2	7.3
DIUTAN GUM With NaCl	Recovery rate [%]	9.5	3.7	4.3

3.3 Oscillation tests

Figure-3A and Figure-3B show the stress sweep test of diutan gum solution (4300 ppm) at 20^0C and 60^0C, respectively. The constant values of the elastic, viscous and complex modulus are an indicator of the linear viscoelasticity range.

Figure-3C and Figure-3D show the stress sweep test of diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 20^0C, and 60^0C, respectively. The constant values of the elastic, viscous and complex modulus are an indicator of the linear viscoelasticity range.

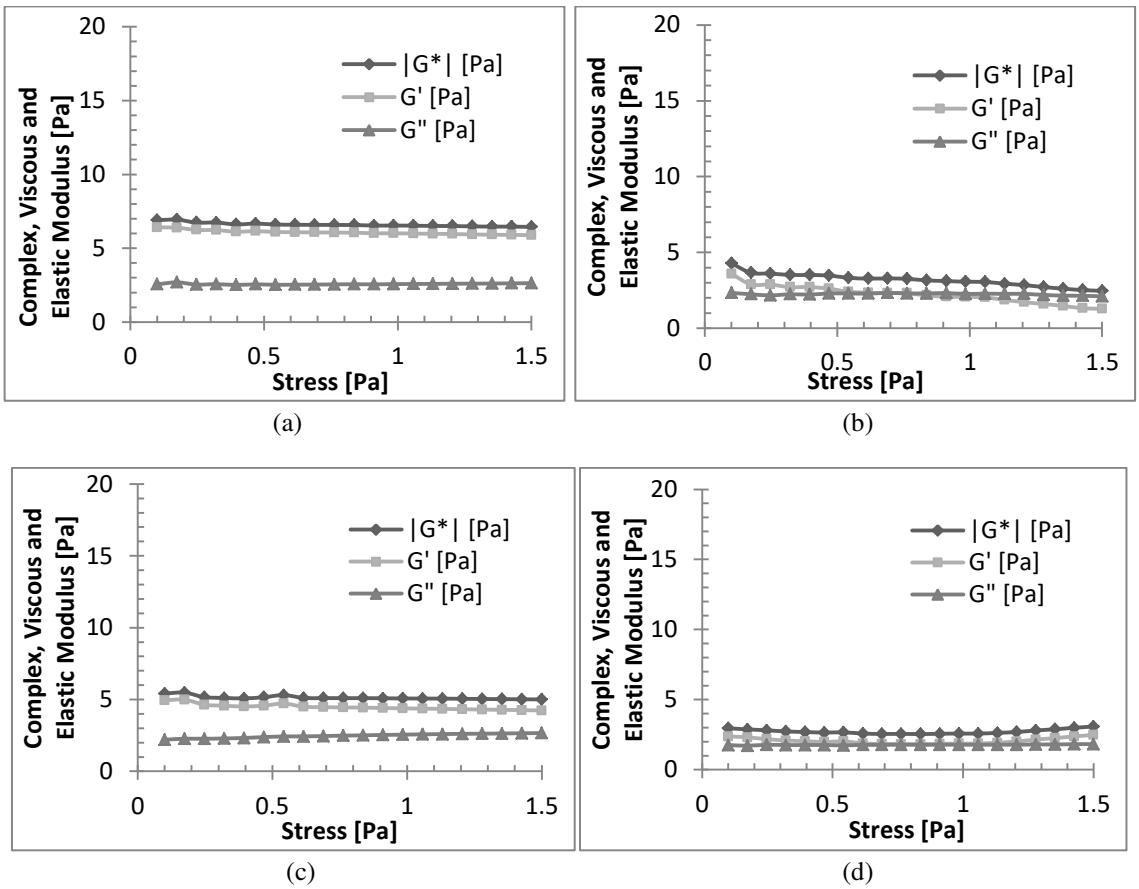


Figure-3. (A) Complex, viscous and elastic modulus of diutan gum solution (4300 ppm) at 1Hz and 20^0C; (B) Complex, viscous and elastic modulus of diutan gum solution (4300 ppm) at 1Hz and 60^0C; (C) Complex, viscous and elastic modulus of diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 1 Hz and 20^0C; (D) Complex, viscous and elastic modulus of diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 1 Hz and 60^0C

Figure-4A and Figure-4B show the frequency sweep test of diutan gum solution (4300 ppm) at 20^0C and 60^0C, respectively. The amplitude of tension was 1 Pa (within the linear viscoelastic region).

At 20°C, the elastic modulus (G ') is greater than viscous modulus (G") in the case of angular velocity higher than 0.7 rad.s^{-1}, evidencing a predominance of elastic effects. The modulus of complex viscosity ($|\eta^*|$) decreases with increasing frequency, as expected.

At 60°C, the elastic modulus (G ') is greater than viscous modulus (G ") in the case of angular velocity higher than 2 rad.s^{-1}, evidencing a predominance of elastic effects. The modulus of complex viscosity ($|\eta^*|$) decreases with increasing frequency, as expected.

Figure-4C and Figure-4D show the frequency sweep test of diutan gum solution (4300 ppm) with NaCl(40000 ppm) at 20^0C, and 60^0C, respectively. The amplitude of tension was 1 Pa (within the linear viscoelastic region).

At 20^0 C, with the salt addition, the elastic modulus (G ') is greater than viscous modulus (G") in the case of angular velocity higher than 0.7 rad.s^{-1}, evidencing a predominance of elastic effects. The modulus of complex viscosity ($|\eta^*|$) decreases with increasing frequency, as expected.

At 60°C, with the salt addition, the viscous modulus (G") keep higher than the elastic modulus (G') in the frequency range investigated, evidencing a predominance of viscous effects. The modulus of complex viscosity ($|\eta^*|$) decreases with increasing frequency, as expected.

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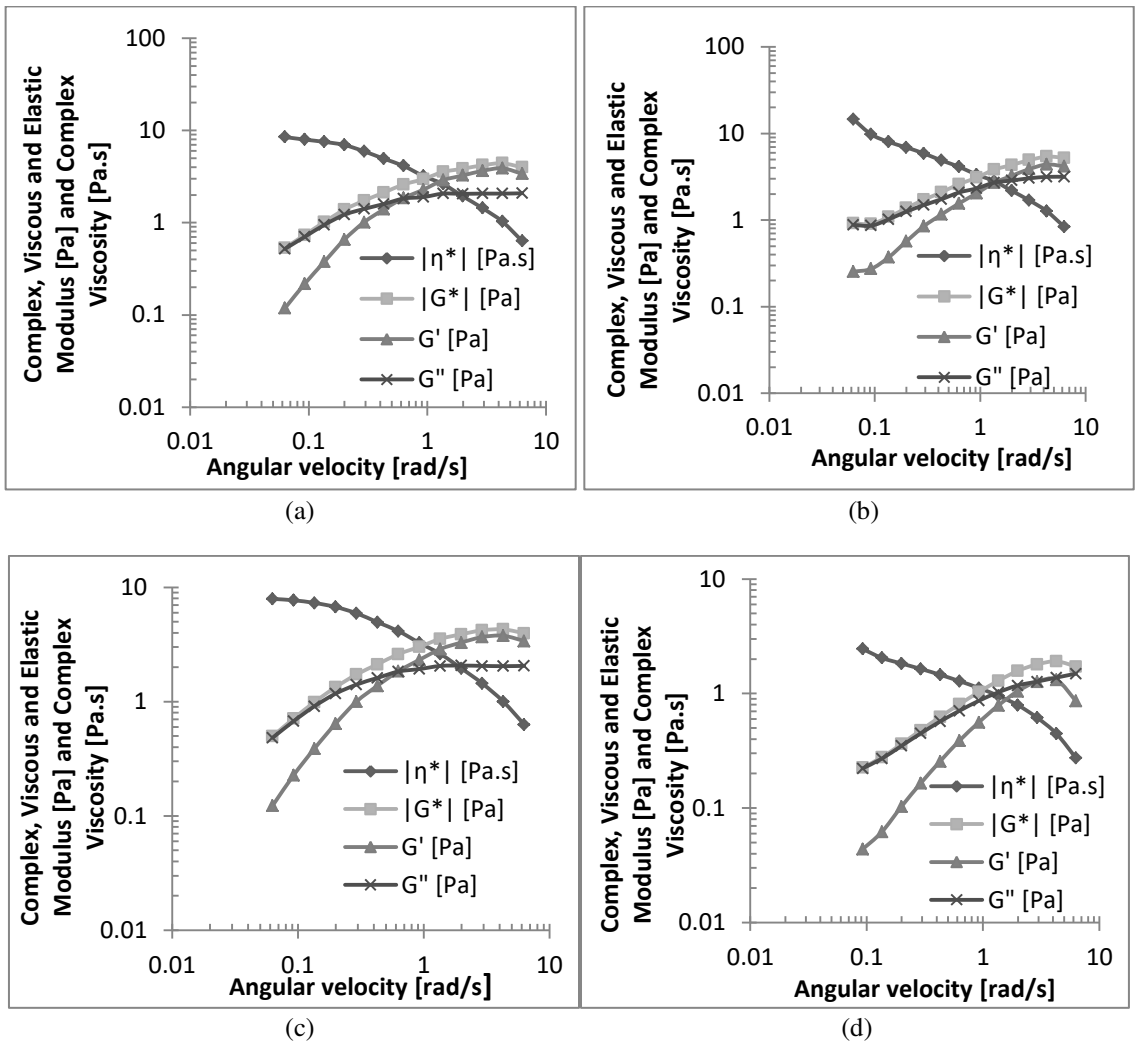

Figure-4. (A) Complex, viscous and elastic modulus and complex viscosity of diutan gum solution (4300 ppm) at 1 Pa and 20^0C; (B) Complex, viscous and elastic modulus and complex viscosity of diutan gum solution (4300 ppm) at 1 Pa and 60^0C; (C) Complex, viscous and elastic modulus and complex viscosity of diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 1 Pa and 20^0C; (D) Complex, viscous andelastic modulus and complex viscosity of diutan gum solution (4300 ppm) with NaCl (40000) at 1 Pa and 60^0C

The G' and G'' modules can be presented as a function of frequency through the power law model (Rao, 1999; Özkan*et al*., 2002):

$$G' = k'\omega^{n'} \quad (3)$$

$$G'' = k''\omega^{n''} \quad (4)$$

where: k ' and k " are constants and n' and n" are the exponents of angular velocity (ω). The values obtained for k ', k ", n' and n" are found in Table-2.

As described by Rao (1999) the data of ln (G', G") vs ln w is subject to linear regression, being n' and n" the magnitudes of slop and k ' and k " the intercepts(Chang *et al*., 2004).

The values obtained for k ', k ", n' and n" of diutan gum solution (4300ppm) and diutan gum solution (4300 ppm) with NaCl (40000 ppm) are shown in Table 3.

At 20°C and 60°C, in the case of diutan gum with and without NaCl, the G' shows a greater dependence on angular velocity than G" (n'› n").

At the temperature of 20° C, the values of k 'and k "and n' and n" are quite similar in the case of diutan gum with and without NaCl .

At the temperature of 60° C the addition of NaCl results in decrease of the value of k' and k" as compared with the diutan gum without NaCl.

As shown in Table-3 in some cases the obtained values of R^2 were less than 0, 90 indicating a not so good fitting data. In this sense, it an exponential model was used to describe the G' and G'' modules as a function of w:

$$G' = a' + b' e^{(w/c,)} \quad (5)$$

$$G'' = a'' + b'' e^{(w/c,,)} \quad (6)$$

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Table-3. Parameters of Power Law model for G'[Pa] and G''[Pa] as a function of w[rad.s^{-1}].

	TEMPERATURE (°C)							
	20				60			
	G' (Pa)		G" (Pa)		G' (Pa)		G" (Pa)	
DIUTAN GUM	k'	2.022	k"	1.665	k'	1.996	k"	2.167
	n'	0.4157	n"	0.2015	n'	0.4940	n"	0.2731
	R^2	0.870	R^2	0.797	R^2	0.9370	R^2	0.9333
DIUTAN GUM With NaCl	k'	2.025	k"	1.656	k'	0.5632	k"	0.8066
	n'	0.4125	n"	0.2035	n'	0.4687	n"	0.3808
	R^2	0.8669	R^2	0.7748	R^2	0.7616	R^2	0.9580

R - CORRELATION COEFFICIENT

Table-4 shows the values of R^2 and the constants: a'; a''; b'; b''; c' and c'' for the exponential model (Equation5 and Equation 6) in the case of diutan gum solution (4300 ppm) and diutan gum solution (4300 ppm) with NaCl (40000 ppm).

As shown in Table-4 the exponential model fits very well (R^2>0.99 for all cases, except one where R^2>0.92) the data of the curves G' and G'' as a function of w

Table-4. Parameters of Exponential model for G'[Pa] and G''[Pa] as a function of w[rad.s^{-1}].

	TEMPERATURE (°C)							
	20				60			
	G' (Pa)		G" (Pa)		G' (Pa)		G" (Pa)	
DIUTAN GUM	a'	3.665	a"	2.051	a'	4.406	a"	3.126
	b'	-3.816	b"	-1,847	b'	-4.395	b"	-2.467
	c'	-0.8544	c"	-0.2832	c'	-1.434	c"	-0.7676
	R^2	0.9926	R^2	0.9934	R^2	0.9966	R^2	0.9970
DIUTAN GUM With NaCl	a'	3.643	a"	2.045	a'	1.172	a"	1.433
	b'	-3.808	b"	-1.924	b'	-1.299	b"	-1.282
	c'	-0.8318	c"	-0.2691	c'	-1.062	c"	-1.174
	R^2	0.9950	R^2	0.9970	R^2	0.9270	R^2	0.9952

R - CORRELATION COEFFICIENT

3.4 Structural recovery tests

Figure-5A and Figure-5B show the results for the structural recovery of diutan gum at 20^{0}C and 60^{0}C, respectively. At20^{0}C the average viscosity at the first stage of shear was 1.494 Pa. sand the average viscosity at the final stage of shear was 1.528Pa.s, resulting in a recovery percentage of 102.2% (($\eta_{final\ stage}/\eta_{first\ stage}$) x100). At 60^{0}C the average viscosity at the first stage of shear was 1.119Pa.s and the average viscosity at the final stage of shear was 1.066Pa.s, resulting in a recovery percentage of 95.3%.

In the case of diutan gum with NaCl, Figure 5C and Figure 5D show the results for the structural recovery at 20^{0}C and 60^{0}C, respectively. At20^{0}C the average viscosity at the first stage of shear was 1.131 Pa.s and the average viscosity at the final stage of shear was 1.134Pa.s, resulting in a recovery percentage of 100.2%. At 60^{0}C the average viscosity at the first stage of shear was 1.009 Pa.s and the average viscosity at the final stage of shear was 0.960Pa.s, resulting in a recovery percentage of 95.1%.

It can be noted that the increase in the temperature results in a decrease in the recovery percentage of diutan gum solutions.

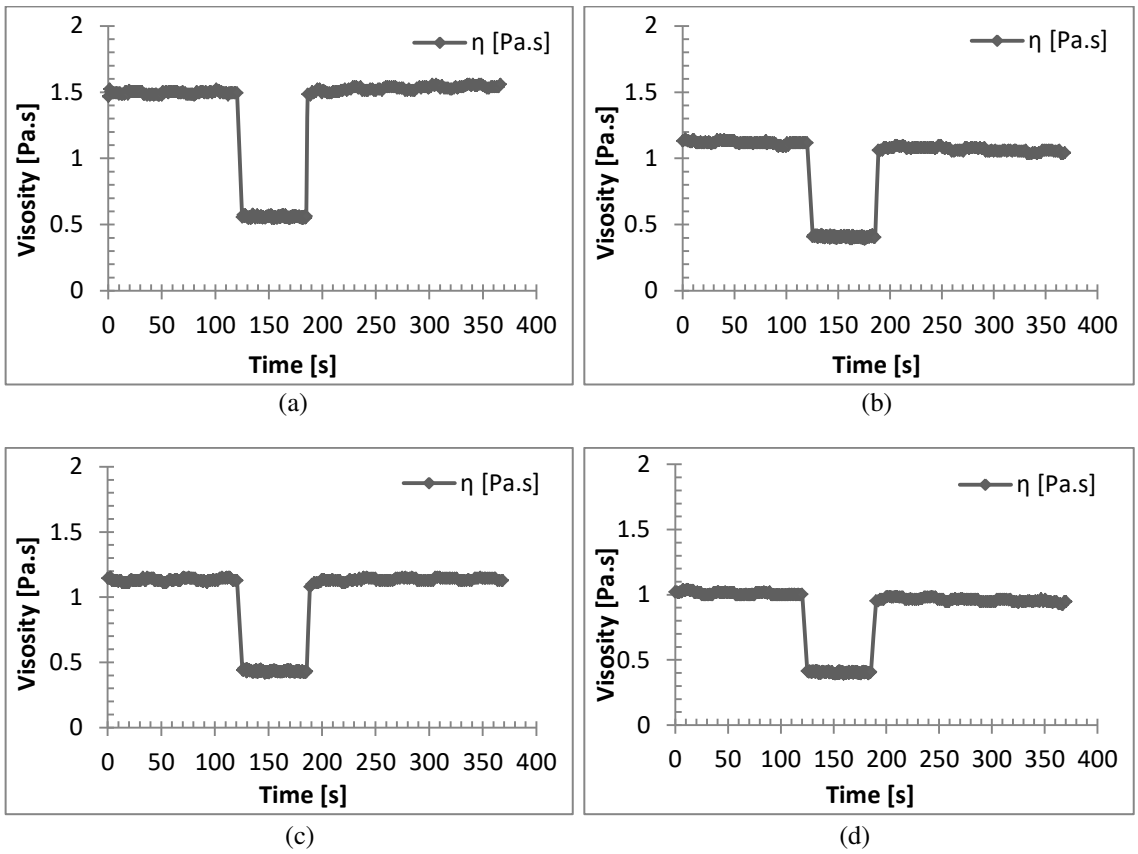

Figure-5. (A) Structural recovery of diutan gum solution (4300 ppm) at 20^0C (5 s^{-1} during 120 s; 15 s^{-1} during 60s and 5 s^{-1} during 180s); (B) Structural recovery of diutan gum solution (4300 ppm) at 60^0C (5s^{-1} during 120 s; 15 s^{-1} during 60s and 5 s^{-1} during 180s); (C) Structural recovery of diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 20^0C (5 s^{-1} during 120 s; 15 s^{-1} during 60s and 5 s^{-1} during 180s); (D) Structural recovery of diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 60^0C (5 s^{-1} during 120 s; 15 s^{-1} during 60s and 5 s^{-1} during 180s)

Wang *et al.* (2009) also used the in-shear structural recovery test in order to investigate the capability of the pastes (waxy maize starch and xanthan gum mixtures in the presence of sucrose) to recover their original structure under low shear conditions after decomposition under high-shear conditions. Achayuthakan and Suphantharika (2008) used the in-shear structural recovery test in the case of waxy corn starch/guar gum and waxy corn starch/ xanthan gum pastes.

3.5 Cox-Merzrule

Cox-Merzrule (Cox and Merz, 1958), proposed that the viscosity (η) should be the same function of shear rate ($\dot{\gamma}$) as the modulus of complex viscosity $|\eta^*|$ is of angular velocity (w) (Barnes *et al*., 1989).

Figure 6Aand Figure6Bshow the viscosity and complex viscosity of diutan gum solution (4300 ppm) at 20^0C and 60^0C respectively. At 60^0C the Cox-Merz rule is better applicable.

Figure-6C and Figure-6D show the viscosity and complex viscosity of diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 20^0C and 60^0C respectively. In this case, the Cox-Merzrule is better applicable at 20^0C.

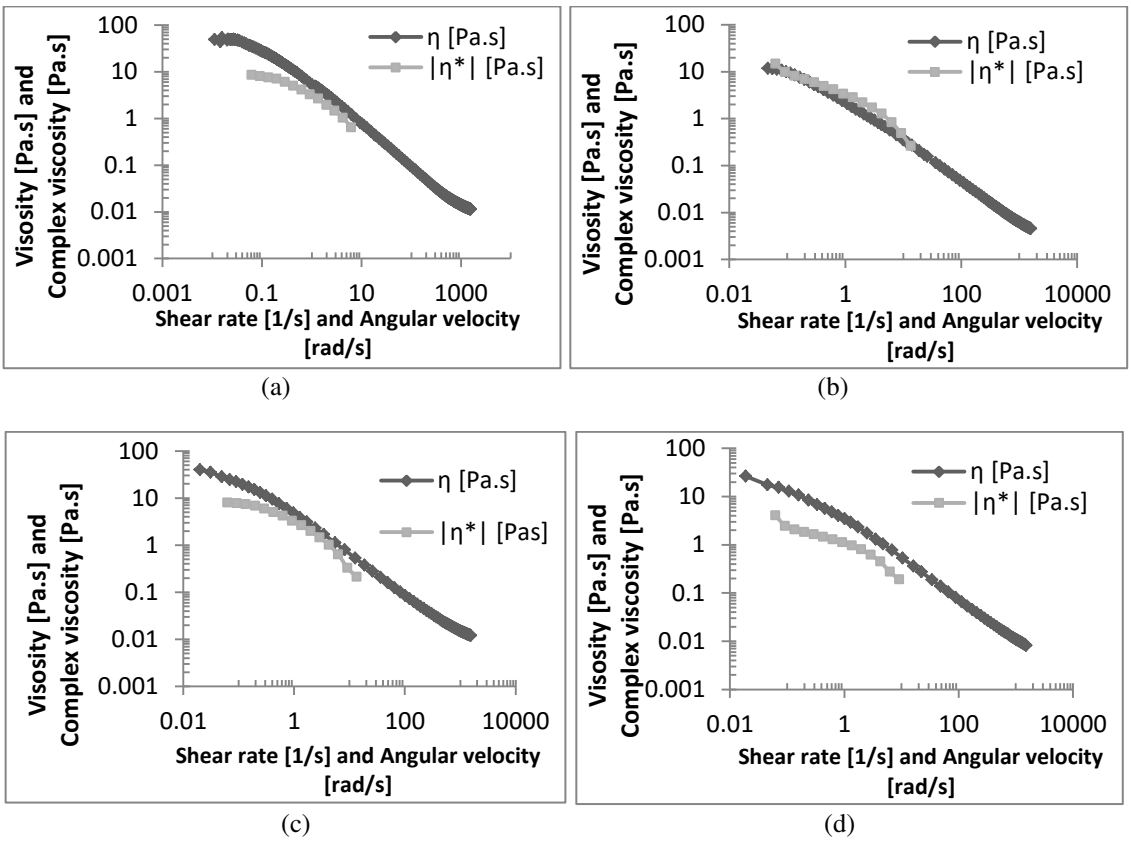

Figure-6. (A) Viscosity and Complex Viscosity of diutan gum solution (4300 ppm) at 20^0C; (B) Viscosity and Complex Viscosity of diutan gum solution (4300 ppm) at 60^0C; (C) Viscosity and Complex Viscosity of diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 20^0C; (D) Viscosity and Complex Viscosity of diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 60^0C.

CONCLUSIONS

Aqueous diutan gum solution showed a pseudoplastic and viscoelastic behavior, even with salt addition.

The viscosity of aqueous diutan gum solution changes very litle in the presence of NaCl. The Power-Law model fits well (R^2> 0, 96) the pseudoplastic behavior of aqueous diutan gum solutions with and without NaCl.

As shown in the creep and recovery test, the recovery is more accentuated in the case of diutan gum without NaCl. At higher temperature, the deformation (strain) is greater. The recovery rate variated from 7.3% to 30.6% in the case of diutan gum solution (4300 ppm) in the temperature range of 60^0C to 20^0Cand from 4.3% to 9.5% in the case of diutan gum solution (4300 ppm) with NaCl (40000ppm) in the temperature range of 60^0C to 20^0C.

With the salt addition, at 60^0C, the viscous modulus (G") keep higher than the elastic modulus (G') in the frequency range investigated. In the others cases, for angular velocities greater than an specific value, the elastic modulus keep higher than viscous modulus indicating a gel behavior of diutan aqueous solutions.

The Exponential model fits very well (R^2> 0, 99, except one case that R^2 = 0, 9270) the viscoelastic (G'(w), G"(w)) behavior of aqueous diutan gum solutions with and without NaCl.

The structural recovery test shows that the increase in the temperature results in a decrease in the recovery percentage of diutan gum solutions with and without NaCl.

The Cox-Merz rule is better applicable in the case of diutan gum solution (4300 ppm) at higher temperature (60^0C).

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