International Journal of Sciences

Research Article Volume 8 – May 2019 (05)

The Influence of Inorganic Cations Addition on Rheology of Aqueous Diutan Gum Solutions

Roberto Guimarães Pereira[1] ✎, Guilherme Pereira Mota[2], Ithamar Ribeiro Rangel[3], João Crisósthomo de Queiroz Neto[4]

[1]Fluminense Federal University – UFF/TEM/PGMEC/PGEB/MSG, Rua Passo da Pátria, 156, São Domingos - CEP: 24210-240, Niterói, RJ, Brazil
[2]Fluminense Federal University – UFF/PGMEC, Rua Passo da Pátria, 156, São Domingos - CEP: 24210-240, Niterói, RJ, Brazil
[3]Geraldo Di Biase University Centre, Rua Dep.Geraldo Di Biase, 81, Volta Redonda-RJ, Brazil
[4]Fluminense Federal University – UFF/ Petroleum Engineering Department, Rua Passo da Pátria, 156, São Domingos - CEP: 24210-240, Niterói, RJ, Brazil

Abstract: The Influence of cations addition (Na^+, Ca^{2+} and Mg^{2+}) on rheology of aqueous diutan gum solutions was investigated at the temperatures of 20^0C, 40^0C and 60^0C. It was used a solution of diutan gum (4300 ppm) in deionized water with 40000 ppm of NaCl. In this solution, inorganic cations (300 ppm of calcium; 300 ppm of magnesium and 300 ppm of calcium plus 300 ppm of magnesium) were added. Steady shear, creep-recovery and oscillation tests were performed. All aqueous diutan gum solutions showed a pseudoplastic and viscoelastic behavior. The addition of calcium and magnesium cations leads to a gel behavior of the solutions for all cases in the angular velocity range investigated, as demonstrated by mechanical spectra. The recovery rate in the case of aqueous diutan gum solutions with calcium and magnesium is much higher than the recovery rate in the case of aqueous diutan gum solutions with NaCl.

Keywords: Diutan Gum, Rheological Tests, Viscosity, Viscoelasticity, Power-Law Model, Exponential Model, Cox-Merz Rule

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 [1-13]. They are biocompatible, biodegradable, and environmentally Friendly [2, 4, 9].

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 two saccharide L-rhamnopyranosyl side-chain attached to the (1→4) linked glucopyranosyl residue [2,14]. 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 [15]; 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 [16]. Diutan gum acts as: stabilizing; thickening, binding; emulsifying and suspending agent [2].

Few works about diutan gum have been reported in the literature. Some of them are detailed as follows.
Garcia et al. [1] studied the effect of temperature and shear on the microstructure of diutan gum aqueous solution. The viscoelastic moduli were obtained as a function of the temperature for 0.5 wt% diutan gum aqueous solutions. As both stress and temperature increase a decrease in the viscoelastic properties occurred. Diutan gum aqueous solutions at 0.5 wt% exhibit a typical weak-gel behavior. Under flow, they show a shear thinning behavior which was fitted to the Carreau model. Both temperature and stress decrease the molecular interactions and, therefore, the viscoelasticity.

Published at: http://www.ijsciences.com/pub/issue/2019-05/
DOI: 10.18483/ijSci.2077; Online ISSN: 2305-3925; Print ISSN: 2410-4477

✎ Roberto Guimarães Pereira (Correspondence)
☎ + 55 21 2629-5419

Li et al. [16] 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. [2] 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 gel properties 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 et al. [15] 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. [14] 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 [17] 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 welan gum which could be attributed to the molecular weight and to the long-side chain of diutan gum leading to greater entanglement and intertwining.

The literature about diutan gum is scarce and there is a gap concerning the influence of inorganic cations addition on aqueous diutan gum solution.

In this paper, it was investigated the influence of cations addition (Na^+, Ca^{2+} and Mg^{2+}) 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 (4300 ppm) in deionized water with 40000 ppm of NaCl. In this solution, inorganic cations (300 ppm of calcium; 300 ppm of magnesium and 300 ppm of calcium plus 300 ppm of magnesium) were added. The NaCl was used to simulate the marine environment and the cations of calcium and magnesium were inserted to simulate, for example, contamination by the existing cement in the oil wells. 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 was applicable in the case of the aqueous diutan gum solutions with NaCl in the presence of the cations of calcium and magnesium.

2. Materials and methods

2.1. Sample preparation

The diutan gum was manufactured by CP Kelco. The NaCl, $CaCl_2$ and $MgCl_2$ were manufactured by Anidrol Produtos para Laboratórios Ltda - Brazil.
The cations of sodium (Na^+), calcium (Ca^{2+}) and magnesium (Mg^{2+}) were added in the aqueous diutan gum solutions.

Four different aqueous diutan gum solutions were used in the tests: (i) diutan gum (4300 ppm) added as a powder in deionized water with 40000 ppm of NaCl; (ii) diutan gum (4300 ppm) added as a powder in deionized water with 40000 ppm of NaCl and 300 ppm of calcium; (iii) diutan gum (4300 ppm) added as a powder in deionized water with 40000 ppm of NaCl and 300 ppm of magnesium; (iv) diutan gum (4300 ppm) added as a powder in deionized water with 40000 ppm of NaCl and 300 ppm of calcium and 300 ppm of magnesium The solutions stayed under mechanical shaking at room temperature of 25^{0}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^{0}C. 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 as 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.

3. Results and discussion

3.1. Steady shear tests

Figures 1, 2 and 3 present the flow curves at 20ºC, 40ºC and 60ºC, comparing the behavior of the viscosity with respect to the shear rate. Samples of aqueous diutan gum solutions were subjected to a stress ranging from 0.5 to 30 Pa.

It is evident the pseudoplastic behavior presented by the samples, characterized by the decrease of the viscosity in relation to the increase of shear rate. The addition of the inorganic cations caused a slight change in the curve. The presence of the cations of calcium and magnesium in the aqueous diutan gum solutions with NaCl caused an increase in viscosity.

The pseudoplastic behavior of diutan gum solution with and without NaCl is also reported at the literature [2, 16].
The decrease in the apparent viscosity with the increase in the shear rate is due to the fact the shear deforms and disaggregates the entanglements, causing their alignment in the flow direction which reduces the hydrodynamic drag and, therefore, the viscosity [1].

The pseudoplastic behavior of fluids is of great interest, for example, because it facilitates pumping [1].
Xu et al. [2] reported that the apparent viscosity of aqueous diutan gum solution changes negligibly in the presence of inorganic salts.

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) with NaCl (4000ppm) and calcium (300ppm); aqueous diutan gum solution (4300 ppm) with NaCl (4000ppm) and magnesium (300ppm) and aqueous diutan gum solution (4300 ppm) with NaCl (4000ppm) and calcium (300ppm) and magnesium (300ppm), for shear rate less than 0.04s^{-1}, at 40^{0}C and 60^{0}C (Figures 2 and 3).
Garcia et al. [1] also pointed out a Newtonian viscosity at low shear rates in steady shear flow curves of diutan gum aqueous solutions at different temperatures and at 0.5 wt%. In this region, the rates of formation and breakdown of macromolecular entanglements are similar. For this reason, the viscosity remains constant.

Some fluids at higher shear rate reach another Newtonian plateau (η_∞), but in this study it was not observed. A complete alignment of diutan gum macromolecules at higher shear rate was not observed.

Table 1 shows the parameters of the Power Law Model (Equation 1) for the diutan gum solution (4300 ppm) with NaCl (40000ppm), calcium (300ppm) and magnesium (300ppm) at 20^{0}C, 40^{0}C and 60^{0}C.

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

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

The Power-Law model fits very well (R^2 > 0,99) the pseudoplastic behavior of aqueous diutan gum solutions with NaCl, calcium and magnesium in the range of shear rate from 0.1s^{-1} to 10000s^{-1}.

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.[18] in the case of welan gum and xanthan gum; Marcotte et al. [19] in the case of xanthan gum and Wang et al. [20] in the case of xanthan gum.

3.2. Creep and recovery tests

As shown in Figures 4 to 9 the recovery is more accentuated in the case of: diutan gum with NaCl and calcium; diutan gum with NaCl and magnesium and diutan gum with NaCl and calcium and magnesium. The presence of the cations of calcium and magnesium in the aqueous diutan gum solution promotes a higher recovery when compared with the case of aqueous diutan gum with NaCl. This recovery of the aqueous diutan gum solutions is an indicator of the sample viscoelastic behavior. Table 2 shows the recovery rate of aqueous diutan gum solutions with NaCl, calcium and magnesium 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:
Recovery rate [%] = $[(\gamma_T - \gamma_P)/\gamma_T] \times 100$ (2)

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

The values of recovery rate in the case of aqueous diutan gum solutions with calcium and magnesium range from 36.7% to 91.3%, much higher than the recovery rate in the case of aqueous diutan gum solutions with NaCl that ranged from to 4.8 to 9.5%, at the temperatures of 20^0C, 40^0C and 60^0C. The addition of the cations of calcium and magnesium enhanced the viscoelasticity of the aqueous diutan gum solucion with NaCl.

The recovery rate (Equation 2) has also been used by Wu et al. [21] to identify the recovery percentage of extruded flaxseed-maize pastes.

3.3. Oscillation tests

Figures 10 to 21 show the results of the oscillation tests (frequency ramp in the range of 0.01 to 20 rad.s^{-1}) with constant stress of 1 Pa (within the linear viscoelasticity range) of the aqueous diutan gum solutions at temperatures of 20ºC and 60ºC.

Figures 11 to 13 show the values of the elastic, viscous and complex modulus and the complex viscosity of the aqueous diutan gum solution with NaCl in the presence of the cations of calcium, magnesium and calcium plus magnesium, at the temperature of 20^0C, respectively. In the mechanical spectra, a gel behavior of the solutions (G'>G") is observed for all cases, in the angular speed range investigated. There was a predominance of the elastic component (G') over the viscous component (G").

Figure 10 shows the values of the elastic, viscous and complex modulus and the complex viscosity of the aqueous diutan gum solution with NaCl in the absence of the calcium and magnesium cations at the temperature of 20^0C. In this case, the predominance of the elastic effects (G'>G") occurs only for angular velocity values greater than 0.7 rad.s^{-1}. Mechanical spectra showed a crossover point between G' and G".

Figures 15 to 17 show the values of the elastic, viscous and complex modulus and the complex viscosity of the aqueous diutan gum solution with NaCl in the presence of the cations of calcium, magnesium and calcium plus magnesium, at the temperature of 60^0C, respectively. A gel behavior of the solutions (G'>G") is observed for all cases, in the angular speed range investigated.

Figure 14 shows the values of the elastic, viscous and complex modulus and the complex viscosity of the aqueous diutan gum solution with NaCl in the absence of the calcium and magnesium cations at the temperature of 60^0C. In this case, it is not observed a predominance of the elastic effects, in the angular speed range investigated.

The G' and G'' modules can be presented as a function of frequency through the Power Law model [22, 23]:

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

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

where: k ' and k " are constants and n' and n" are the exponents of angular velocity (ω).

As described by Rao [22] 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 [24].

The values obtained for k', k", n' and n" of the Power Law model of the aqueous diutan gum solution (4300ppm) with NaCl (40000ppm), of the aqueous diutan gum solution with NaCl in the presence of the cations of calcium (300ppm), magnesium (300ppm) and calcium (300ppm) plus magnesium (300ppm) are shown in Table 3, in the case of the $R^2>0.7$.

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

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

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

Table 4 shows the values of R^2 and the constants: a'; a''; b'; b''; c' and c'' for the exponential model (Equation 5 and Equation 6) in the case of the aqueous diutan gum solution (4300ppm) with NaCl (40000ppm), of the aqueous diutan gum solution with NaCl in the presence of the cations of calcium (300ppm), magnesium (300ppm) and calcium (300ppm) plus magnesium (300ppm).

As shown in Table 4 the exponential model fits well ($R^2>0.9$ for the most of the cases) the data of the curves G' and G'' as a function of w in the range of 0.01 to 10 rad.s^{-1}

The addition of the cations of calcium and magnesium modified the configuration of the curves causing an increase in the values of the complex modulus and the complex viscosity with respect to aqueous diutan gum solution with NaCl as shown in Figures 18 to 21.

3.4. Cox-merz rule

Cox-Merz rule [25] proposes 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) [26].

Figures 22 to 29 show the viscosity and complex viscosity in the case of the aqueous diutan gum solution (4300ppm) with NaCl (40000ppm), of the aqueous diutan gum solution with NaCl in the presence of the cations of calcium (300ppm), magnesium (300ppm) and calcium (300ppm) plus magnesium (300ppm), at 20^0C and 60^0C respectively. The Cox-Merz rule is better applicable in the case of the aqueous diutan gum solutions in the presence of the cations of calcium and magnesium at 60^0C.

4. Conclusions

The aqueous diutan gum solutions tested showed a pseudoplastic and viscoelastic behavior. The addition of the cations modified the rheological parameters of the gum. The presence of the cations of calcium and magnesium in the aqueous diutan gum solutions with NaCl caused an slight increase in viscosity. With the addition of calcium and magnesium, there was an increase in complex modulus and complex viscosity values relative to aqueous diutan gum solution with NaCl, in the angular velocity range investigated. The addition of calcium and magnesium cations leads to a gel behavior of the solutions (G'>G'') for all cases in the angular velocity range investigated.

The Power-Law model fits very well (R^2 > 0,99) the pseudoplastic behavior of aqueous diutan gum solutions with NaCl, calcium and magnesium in the range of shear rate from $0.1s^{-1}$ to $10000s^{-1}$.

As shown in the creep and recovery test, the values of recovery rate in the case of aqueous diutan gum solutions with calcium and magnesium is much higher than the recovery rate in the case of aqueous diutan gum solutions with NaCl at the temperatures of 20^0C, 40^0C and 60^0C. The addition of the cations calcium and magnesium enhanced the viscoelasticity of the aqueous diutan gum solucion with NaCl.

A gel behavior (G'>G") of the aqueous diutan gum solution with NaCl in the presence of the cations of calcium, magnesium and calcium plus magnesium, at the temperatures of 20^0C and 60^0C, is observed in the angular speed range investigated.

The exponential model fits well (R^2>0.9 for the most of the cases) the data of the curves G' and G'' as a function of w in the range of 0.01 to 10 $rad.s^{-1}$.

The Cox-Merz rule is better applicable in the case of the aqueous diutan gum solutions in the presence of the cations of calcium and magnesium at 60^0C.

Diutan gum is an important biopolymer and has many applications: it is an effective additive for improving the viscosity and the performance of cement paste; it has a great potential in tertiary oil recovery. Diutan gum acts as: stabilizing; thickening, binding; emulsifying and suspending agent.

The knowledge of the flow behavior of aqueous diutan gum solutions with the addition of inorganic cations can contribute to the improvement of process in which diutan gum is used.

Acknowledgments

The authors Roberto Pereira and Guilherme Mota are grateful to the National Research Council of Brazil, CNPq, for the financial support.

References

1. García MC, Carmona JA, Santos J, Alfaro MC, Muñoz J, Effect of temperature and shear on the microstructure of a microbial polysaccharide secreted by Sphingomonas species in aqueous solution, Int. J. Biol. Macromol. 2018, 118: 2071–2075.
2. Xu L, Gong H, Dong M, Li Y, Rheological properties and thickening mechanism of aqueous diutan gum solution: Effects of temperature and salts, Carbohydr. Polym. 2015, 132: 620-629.
3. Carmona JA, Lucas A, Ramírez P, Calero N, Muñoz J, Nonlinear and linear viscoelastic properties of a novel type of xanthan gum with industrial applications, Rheol Acta. 2015, 54: 993–1001.
4. Chang I, Im J, Prasidhi AK, Cho GC, Effects of Xanthan gum biopolymer on soil strengthening, Constr. Build. Mater. 2015, 74: 65-72.
5. Lee JS, Kim YS, Song KW, Transient rheological behavior of natural polysaccharide xanthan gum solutions in start-up shear flow fields: An experimental study using a strain-controlled rheometer, Korea-Australia Rheology Journal. 2015, 27(3): 227-239.
6. Lee JS, Song KW, Time-dependent rheological behavior of natural polysaccharide xanthan gum solutions in interrupted shear and step-incremental/reductional shear flow fields, Korea-Australia Rheology Journal. 2015, 27(4): 297-307.
7. Kaur V, Bera MB, Panesar PS, Kumar H, Kennedy JF, Welan gum: Microbial production, characterization, and applications, Int. J. Biol. Macromol. 2014, 65: 454–461.
8. Xu L, Xu G, Yu L, Gong H, Dong M, Li Y, The displacement efficiency and rheology of welan gum for enhanced heavy oil recovery, Polym. Adv. Technol. 2014, 25: 122-1129.
9. Khalil M, Jan BM, Herschel-Bulkley rheological parameters of a novel environmentally friendly lightweight biopolymer drilling fluid from xanthan gum and starch, J. Appl. Polym. Sci. 2012, 124: 595-606.
10. Sonebi M, McKendry D, Effect of Mix Proportions on Rheological and Hardened Properties of Composite Cement Pastes, The Open Construction and Building Technology Journal. 2008, 2: 15-23.
11. Marudova-Zsivanovits M, Jilov N, Gencheva E, Rheological Investigation of Xanthan Gum–Chromium Gelation and its Relation to Enhanced Oil Recovery, Journal of Applied Polymer Science, 2007, 103: 160–166.
12. Dolz M, Hernández MJ, Delegido J, Oscillatory measurements for salad dressings stabilized with modified starch, xanthan gum, and locust bean gum J. Appl. Polym. Sci. 2006. 102: 897-903.
13. Gebert MS, Friend DR, Purified guar galactomannan as an

improved pharmaceutical excipient, Pharm. Dev. Technol. 1989, 3: 315-323.

14. Banerjee P, Mukherjee I, Bhattacharya S, Datta S, Moulik SP, Sarkar D, Sorption of water vapor, hydration, and viscosity of carboxymethylhydroxypropyl guar, diutan, and xanthan gums, and their molecular association with and without salts ($NaCl$, $CaCl_2$, $HCOOK$, CH_3COONa, $(NH_4)_2SO_4$ and $MgSO_4$) in aqueous solution, Langmuir. 2009, 25: 11647-11656.
15. Zhang J, Weissinger EA, Peethamparan S, Scherer GW, Early hydration and setting of oil well cement, Cem. Concr. Res. 2010, 40: 1023-1033.
16. Li Y, Xu L, Gong H, Ding B, Dong M, Li Y, A microbial exopolysaccharide produced by sphingomonas species for enhanced heavy oil recovery at high temperature and high salinity, Energy Fuels. 2017, 31: 3960-3969.
17. Sonebi M, Rheological properties of grouts with viscosity modifying agents as diutan gum and welan gum incorporating pulverised fly ash, Cem. Concr. Res. 2006, 36: 1609-1618.
18. Xu L, Xu G, Liu T, Chen Y, Gong H, The comparison of rheological properties of aqueous welan gum and xanthan gum solutions, Carbohydr. Polym. 2013, 92: 516-522.
19. Marcotte M, Hoshahili ART, Ramaswamy HS, Rheological properties of selected hydrocolloids as a function of concentration and temperature, Food Res. Int. 2001, 34: 695-703.
20. Wang B, Wang LJ, Li D, Özkan N, Li SJ, Maoa ZH, Rheological properties of waxy maize starch and xanthan gum mixtures in the presence of sucrose, Carbohydr. Polym. 2009, 77: 472-481.
21. Wu M, Li D, Wang LJ, Özkan N, Mao ZH, Rheological properties of extruded dispersions of flaxseed-maize blend, J. Food Eng. 2010, 98: 480-491.
22. Rao MA, Rheological behavior of processed fluid and semisolid foods. In M. A. Rao (Ed.), Rheology of Fluid and Semisolid Foods, Maryland: Aspen Pub, 1999, 153-218.
23. Özkan N, Xin H, Chen XD, Application of a depth sensing indentation hardness test to evaluate the mechanical properties of food materials, J. Food Sci. 2002, 67(5): 1814-1820.
24. Chang YH, Lim ST, Yoo B, Dynamic rheology of corn starch–sugar composites, J. Food Eng. 2004, 64: 521-527.
25. Cox WP, Merz EH, Correlation of dynamic and steady flow viscosities, J. Polym. Sci. 1958, 2: 619-622.
26. Barnes HA, Hutton JF, Walters K, An introduction to rheology, New York, Elsevier. 1989.

Table 1 Parameters of the Power Law Model for η (Pa.s) as a function of $\dot{\gamma}$ (s^{-1}) of aqueous diutan gum solution (4300 ppm) with NaCl (40000ppm), calcium (300ppm) and magnesium (300ppm) at 20^0C, 40^0C and 60^0C

		TEMPERATURE (^{0}C)		
		20	**40**	**60**
Diutan gum with NaCl	k [Pa.s^n]	5.558	4.734	3.831
	n	0.467	0.275	0.503
	R^2	0.9954	0.9988	0.9922
	Shear rate range [s^{-1}]	$0.001<\dot{\gamma}<10000$	$0.1<\dot{\gamma}<10000$	$0.001<\dot{\gamma}<10000$
Diutan gum with NaCl and Calcium	k [Pa.s^n]	8.881	9.714	5.183
	n	0.344	0.204	0.165
	R^2	0.9950	0.9998	0.9998
	Shear rate range [s^{-1}]	$0.01<\dot{\gamma}<10000$	$0.1<\dot{\gamma}<10000$	$0.1<\dot{\gamma}<10000$
Diutan gum with NaCl and Magnesium	k [Pa.s^n]	6.866	7.041	5.116
	n	0.412	0.319	0.217
	R^2	0.9986	0.9990	0.9998
	Shear rate range [s^{-1}]	$0.01<\dot{\gamma}<10000$	$0.1<\dot{\gamma}<10000$	$0.1<\dot{\gamma}<10000$
Diutan gum with NaCl, Calcium and Magnesium	k [Pa.s^n]	8.808	8.158	5.849
	n	0.304	0.214	0.214
	R^2	0.9988	0.9998	0.9998
	Shear rate range [s^{-1}]	$0.1<\dot{\gamma}<10000$	$0.1<\dot{\gamma}<10000$	$0.1<\dot{\gamma}<10000$

R - correlation coefficient

Table 2 Percentage of recovery of aqueous diutan gum solution (4300 ppm) with NaCl (40000ppm), calcium (300ppm) and magnesium (300ppm) at 20^0C, 40^0C and 60^0C

		TEMPERATURE (^{0}C)		
		20	40	60
Diutan gum with NaCl	Recovery rate [%]	9.5	6.4	4.8
Diutan gum with NaCl and Calcium	Recovery rate [%]	71.2	86.8	58.6
Diutan gum with NaCl and Magnesium	Recovery rate [%]	91.3	75.7	57.3
Diutan gum with NaCl, Calcium and Magnesium	Recovery rate [%]	36.7	42.8	53.9

Table 3 Parameters of Power Law model for G'[Pa] and G''[Pa] as a function of w[rad.s^{-1}] of aqueous diutan gum solution (4300 ppm) with NaCl (40000ppm), calcium (300ppm) and magnesium (300ppm) at 20^0C and 60^0C

	TEMPERATURE (°C)							
	20				60			
	G' (Pa)		G" (Pa)		G' (Pa)		G" (Pa)	
Diutan gum with NaCl	**k'**	-	**k"**	1.635	**k'**	0.557	**k"**	0.802
	n'	-	**n"**	0.186	**n'**	0.477	**n"**	0.363
	R^2	<0.7	**R^2**	0.7760	**R^2**	0.7799	**R^2**	0.9657
	Angular velocity range [rad.s^{-1}]	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.01<w<9	**Angular velocity range [rad.s^{-1}]**	0.01<w<10
Diutan gum with NaCl and Calcium	**k'**	12.89	**k"**	-	**k'**	8.576	**k"**	-
	n'	0.175	**n"**	-	**n'**	0.211	**n"**	-
	R^2	0.7802	**R^2**	<0.7	**R^2**	0.8834	**R^2**	<0.7
	Angular velocity range [rad.s^{-1}]	0.01<w<20	**Angular velocity range [rad.s^{-1}]**	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.01<w<10
Diutan gum with NaCl and Magnesium	**k'**	11.98	**k"**	-	**k'**	10.53	**k"**	6.924
	n'	0.213	**n"**	-	**n'**	0.209	**n"**	0.2038
	R^2	0.8943	**R^2**	<0.7	**R^2**	0.8716	**R^2**	0.9411
	Angular velocity range [rad.s^{-1}]	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.1<w<20	**Angular velocity range [rad.s^{-1}]**	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.01<w<10
Diutan gum with NaCl, Calcium and Magnesium	**k'**	13.88	**k"**	-	**k'**	7.213	**k"**	-
	n'	0.215	**n"**	-	**n'**	0.211	**n"**	-
	R^2	0.9166	**R^2**	<0.7	**R^2**	0.8582	**R^2**	<0.7
	Angular velocity range [rad.s^{-1}]	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.1<w<20	**Angular velocity range [rad.s^{-1}]**	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.01<w<10

R - correlation coefficient

Table 4 Parameters of Exponencial model for G’[Pa] and G’’[Pa] as a function of w[rad.s^{-1}] of aqueous diutan gum solution (4300 ppm) with NaCl (40000ppm), calcium (300ppm) and magnesium (300ppm) at 20^0C and 60^0C

		TEMPERATURE (°C)							
		20				**60**			
		G’ (Pa)	**G” (Pa)**			**G’ (Pa)**	**G” (Pa)**		
Diutan gum with NaCl	**a’**	3.643	**a”**	2.076	**a’**	1.176	**a”**	1.550
	b’	-3.808	**b”**	-1.929	**b’**	-1.271	**b”**	-1.358
	c’	-0.8381	**c”**	-0.2839	**c’**	-1.104	**c”**	-1.501
	R^2	0.9950	**R^2**	0.9906	**R^2**	0.9306	**R^2**	0.9847
	Angular velocity range [rad.s^{-1}]	0.01<w<9	**Angular velocity range [rad.s^{-1}]**	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.01<w<9	**Angular velocity range [rad.s^{-1}]**	0.01<w<10
Diutan gum with NaCl and Calcium	**a’**	17.69	**a”**	-295.7	**a’**	11.9	**a”**	-
	b’	-11.59	**b”**	300.8	**b’**	-8.738	**b”**	-
	c’	-07089	**c”**	-4488	**c’**	-0.699	**c”**	-
	R^2	0.9481	**R^2**	0.7392	**R^2**	0.9813	**R^2**	<0.7
	Angular velocity range [rad.s^{-1}]	0.1<w<20	**Angular velocity range [rad.s^{-1}]**	0.01<w<20	**Angular velocity range [rad.s^{-1}]**	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.1<w<10
Diutan gum with NaCl and Magnesium	**a’**	16.54	**a”**	-114.4	**a’**	14.61	**a”**	9.919
	b’	-12.34	**b”**	119.1	**b’**	-10.50	**b”**	-6.251
	c’	-0.6539	**c”**	-1900	**c’**	-0.7370	**c”**	-1.220
	R^2	0,9829	**R^2**	0.7186	**R^2**	0.9616	**R^2**	0.9866
	Angular velocity range [rad.s^{-1}]	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.1<w<20	**Angular velocity range [rad.s^{-1}]**	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.01<w<10
Diutan gum with NaCl, Calcium and Magnesium	**a’**	19.31	**a”**	2.606	**a’**	10.00	**a”**	3.119
	b’	-14.02	**b”**	2.742	**b’**	-7.558	**b”**	0.6773
	c’	-0.7225	**c”**	-26.79	**c’**	-0.6557	**c”**	-3.107
	R^2	0.9841	**R^2**	0.7795	**R^2**	0.9845	**R^2**	0.9374
	Angular velocity range [rad.s^{-1}]	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.1<w<20	**Angular velocity range [rad.s^{-1}]**	0.01<w<10	**Angular velocity range [rad.s^{-1}]**	0.1<w<20

R - correlation coefficient

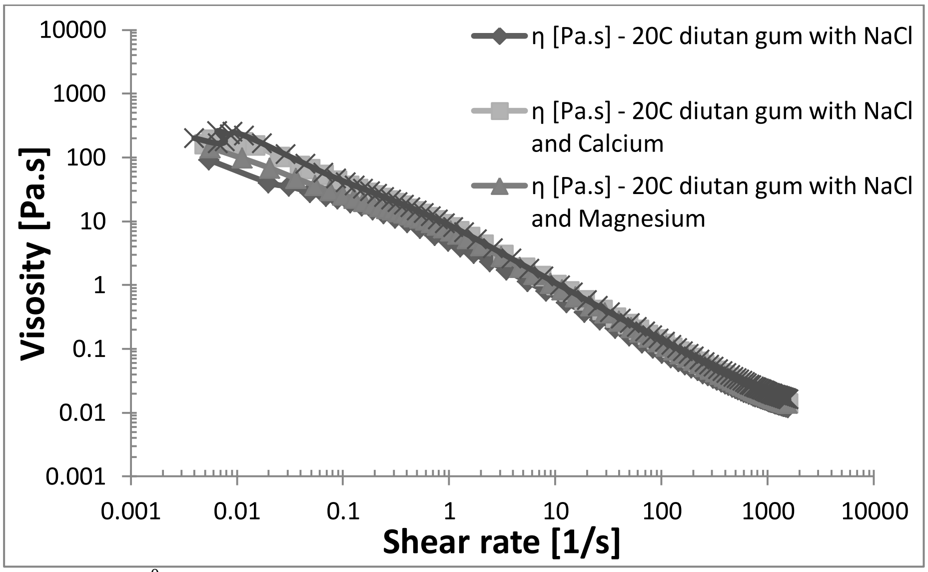

Fig.1 Flow curve at 20^{0}C of aqueous diutan gum solution (4300ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm)

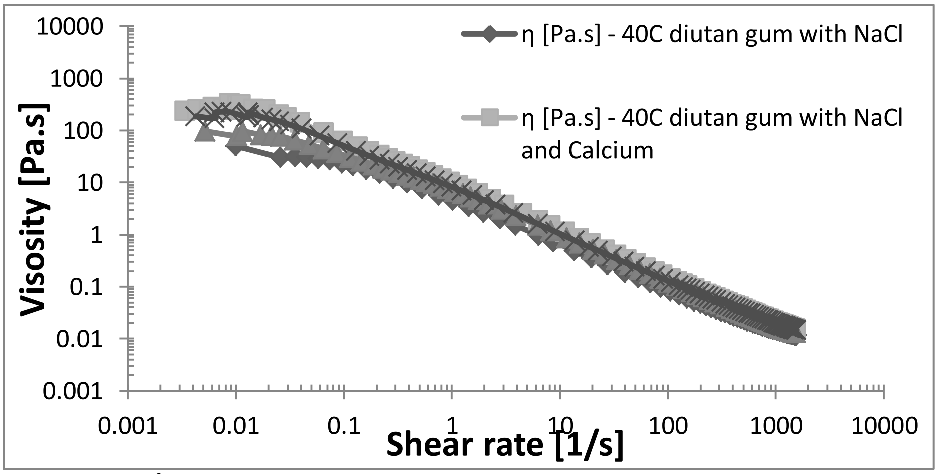

Fig.2 Flow curve at 40^{0}C of aqueous diutan gum solution (4300ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm)

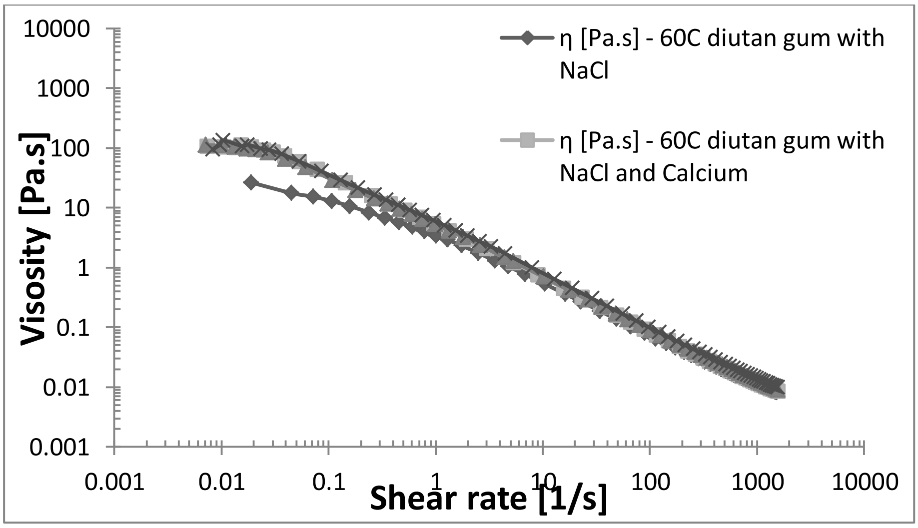

Fig.3 Flow curve at 60^{0}C of aqueous diutan gum solution (4300ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm)

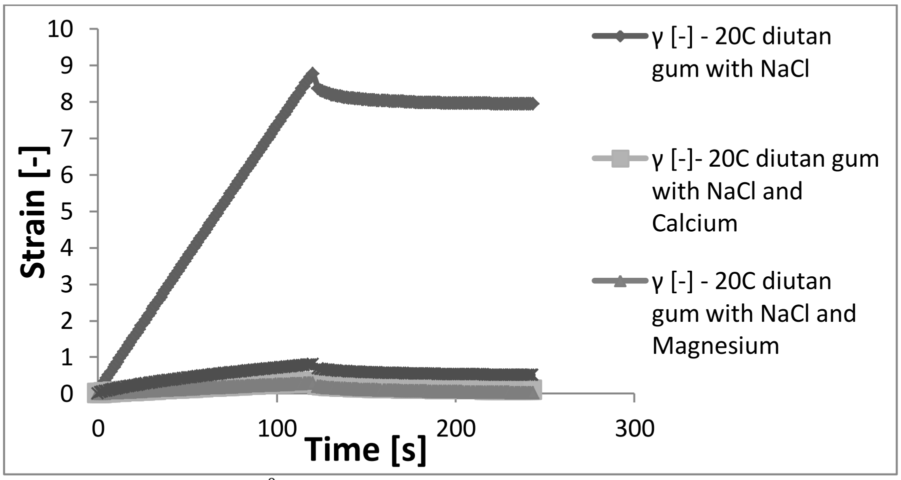

Fig. 4 Creep and recovery at 20^{0}C of aqueous diutan gum solution (4300ppm) with NaCl (40000 ppm) and of aqueous diutan gum solution with NaCl and Calcium (300 ppm) and Magnesium (300 ppm)

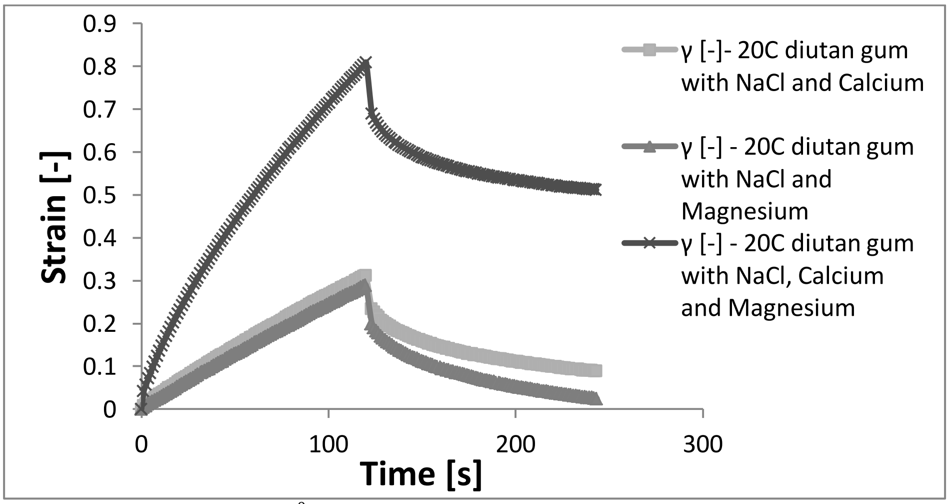

Fig. 5 Creep and recovery at 20^{0}C of aqueous diutan gum solution (4300ppm) with NaCl (40000 ppm) and Calcium (300 ppm) and Magnesium (300 ppm)

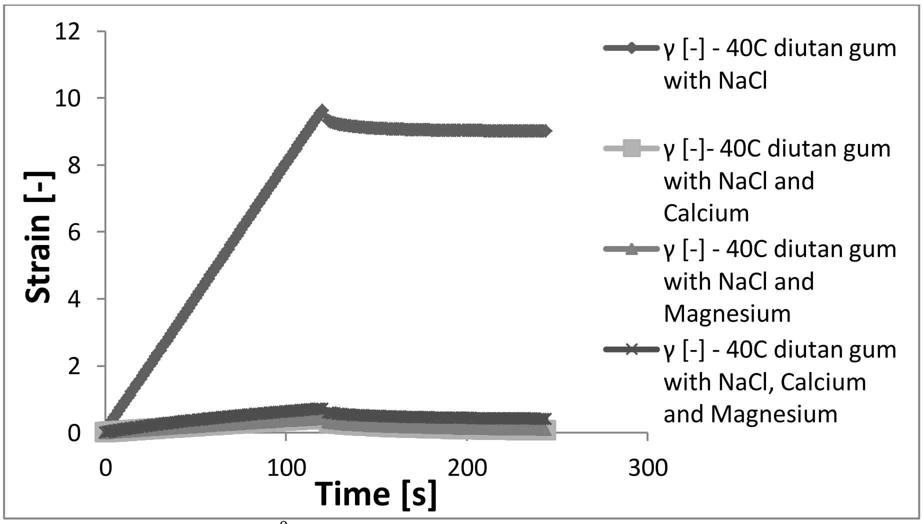

Fig. 6 Creep and recovery at 40^{0}C of aqueous diutan gum solution (4300ppm) with NaCl (40000 ppm) and of aqueous diutan gum solution with NaCl and Calcium (300 ppm) and Magnesium (300 ppm)

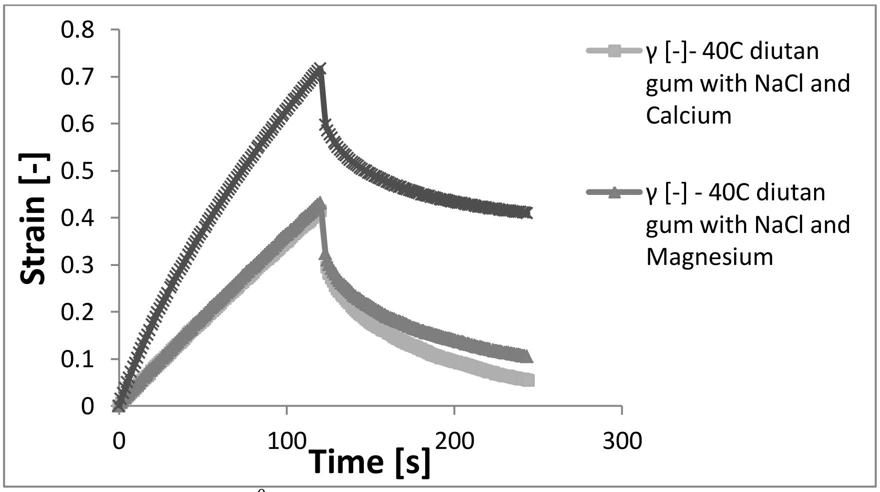

Fig. 7 Creep and recovery at 40^{0}C of aqueous diutan gum solution (4300ppm) with NaCl (40000 ppm) and Calcium (300 ppm) and Magnesium (300 ppm)

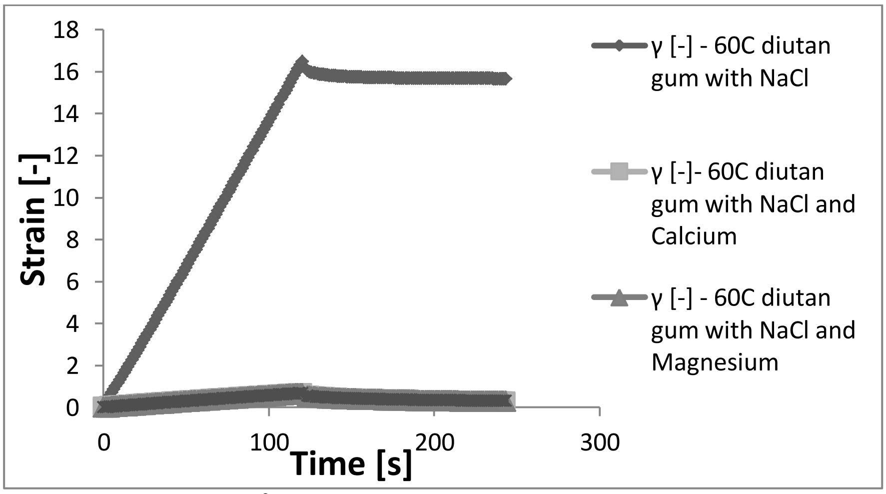

Fig. 8 Creep and recovery at 60^{0}C of aqueous diutan gum solution (4300ppm) with NaCl (40000 ppm) and of aqueous diutan gum solution with NaCl and Calcium (300 ppm) and Magnesium (300 ppm)

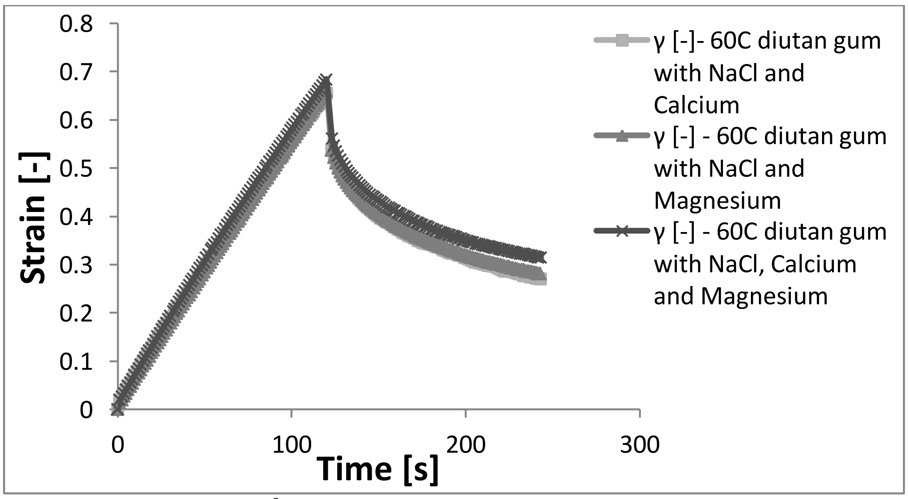

Fig. 9 Creep and recovery at 60^{0}C of aqueous diutan gum solution (4300ppm) with NaCl (40000 ppm) and Calcium (300 ppm) and Magnesium (300 ppm)

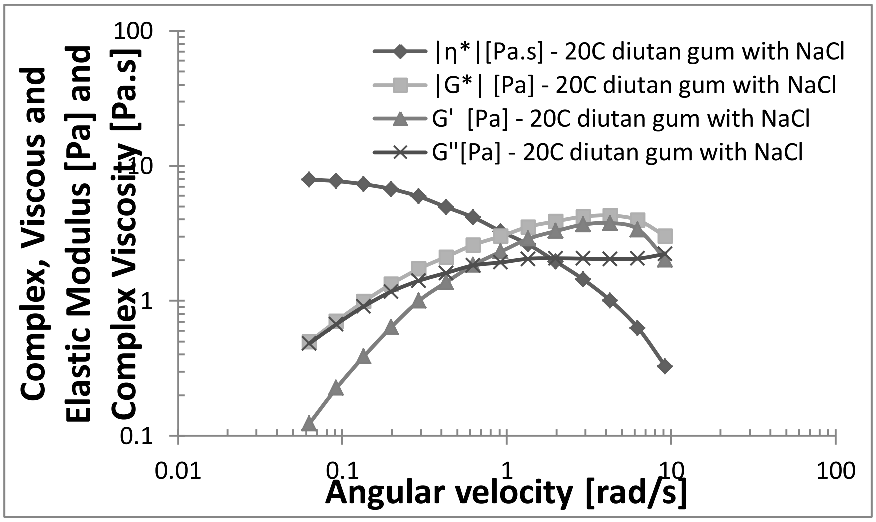

Fig.10 Complex, viscous and elastic modulus and complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 1 Pa and 20^{0}C

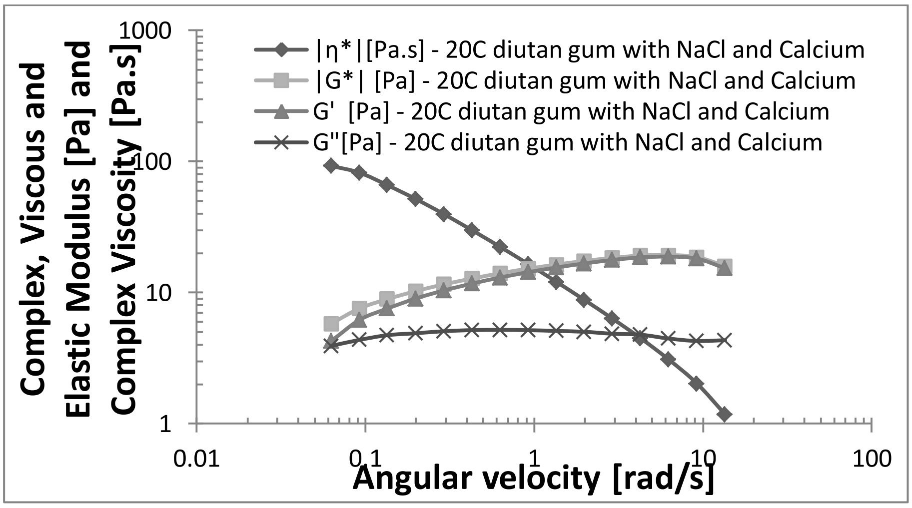

Fig. 11 Complex, viscous and elastic modulus and complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) and Calcium (300 ppm) at 1 Pa and 20^{0}C

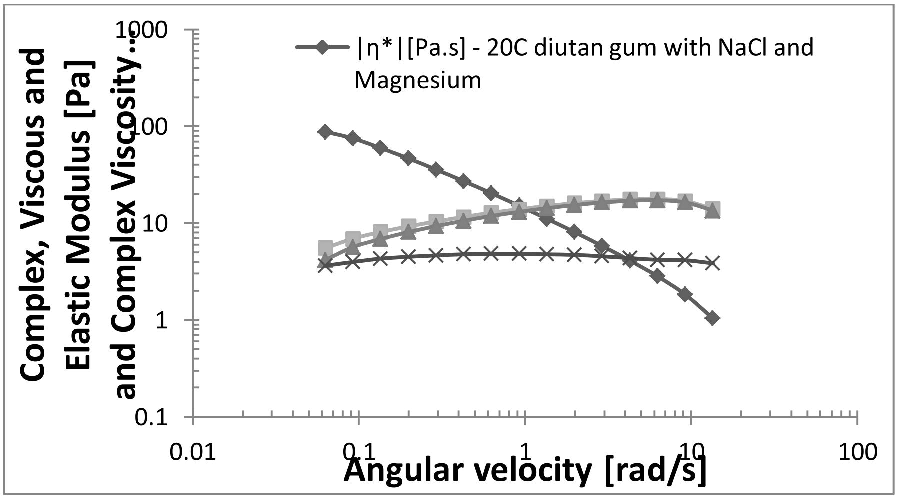

Fig. 12 Complex, viscous and elastic modulus and complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) and Magnesium (300 ppm) at 1 Pa and 20^{0}C

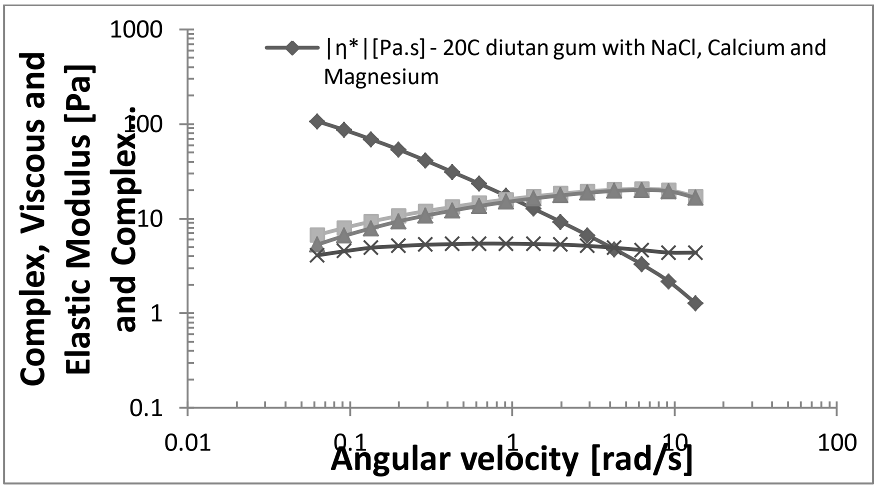

Fig. 13 Complex, viscous and elastic modulus and complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm) at 1 Pa and 20^0C

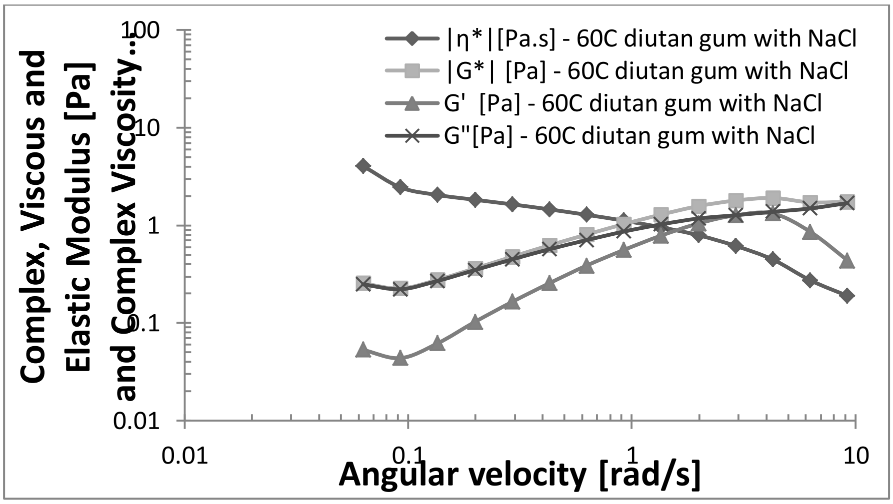

Fig. 14 Complex, viscous and elastic modulus and complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 1 Pa and 60^0C

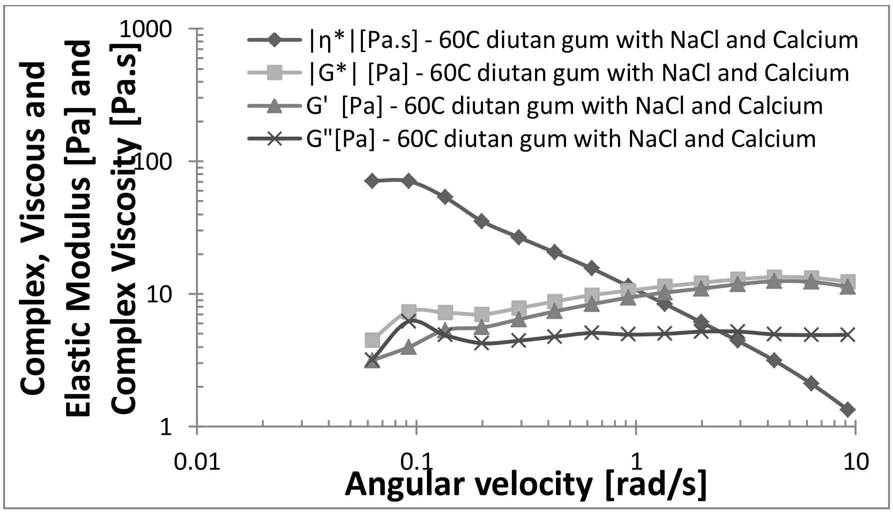

Fig. 15 Complex, viscous and elastic modulus and complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) and Calcium (300 ppm) at 1 Pa and 60^0C

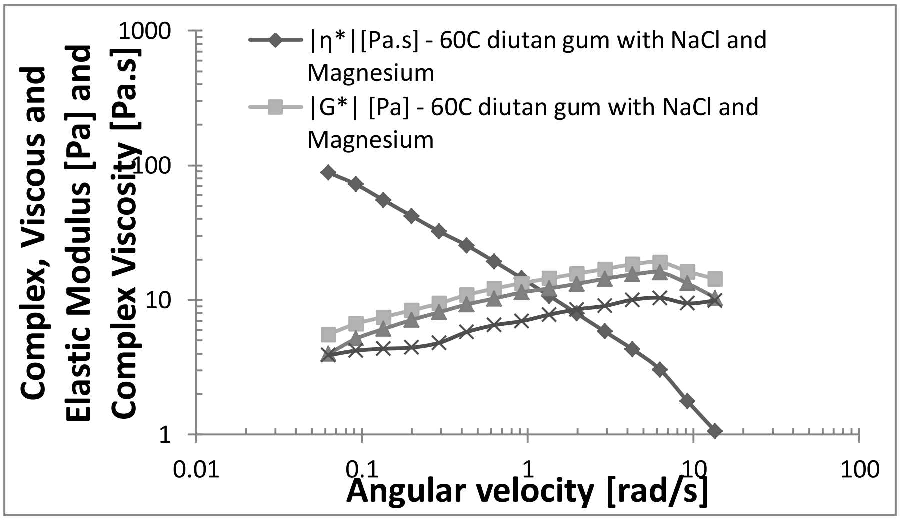

Fig. 16 Complex, viscous and elastic modulus and complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) and Magnesium (300 ppm) at 1 Pa and 60^0C

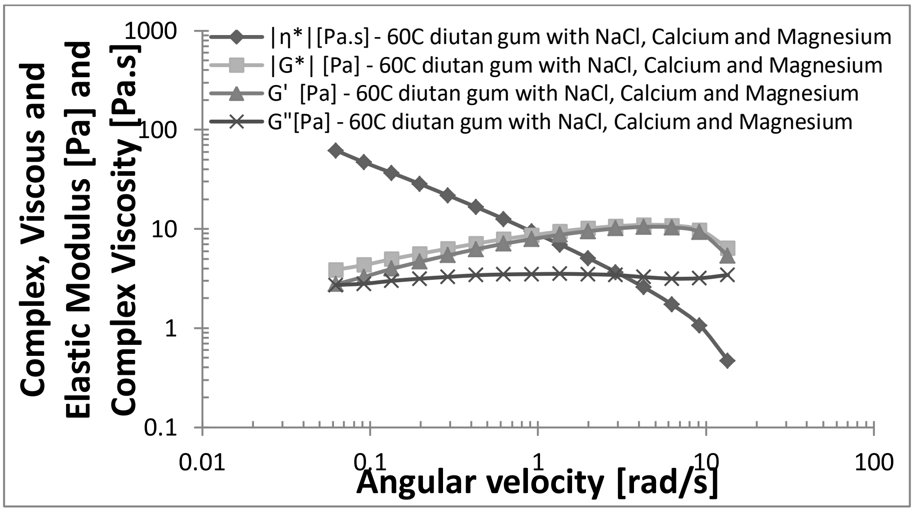

Fig. 17 Complex, viscous and elastic modulus and complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm) at 1 Pa and 60^0C

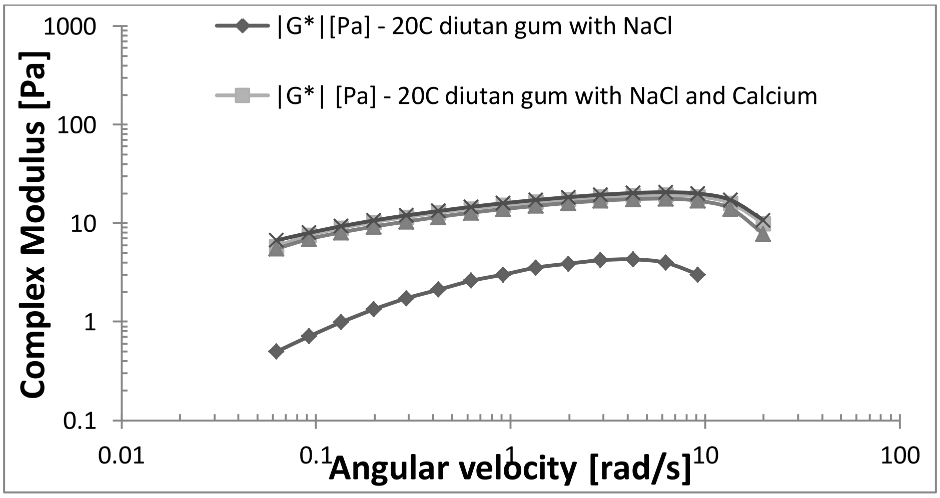

Fig. 18 Complex modulus of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm) at 1 Pa and 20^0C

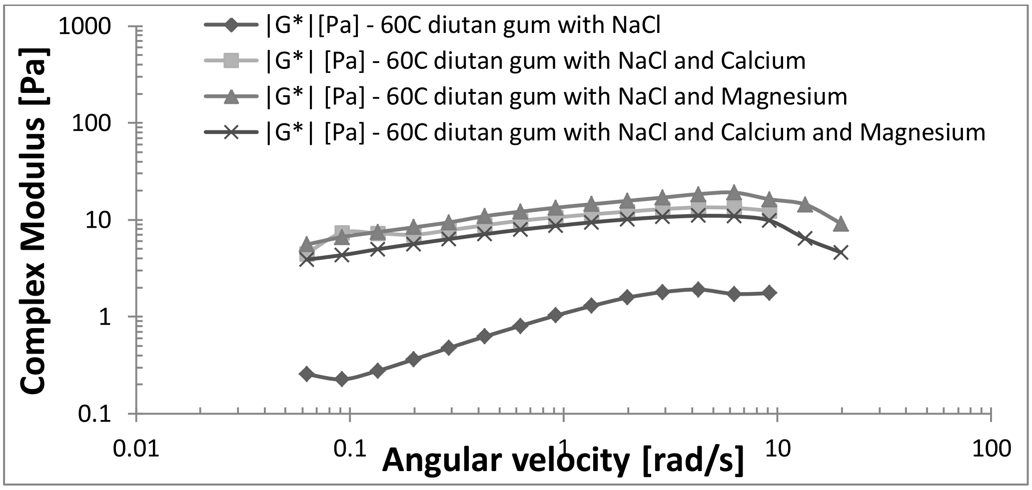

Fig. 19 Complex modulus of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm) at 1 Pa and 60^0C

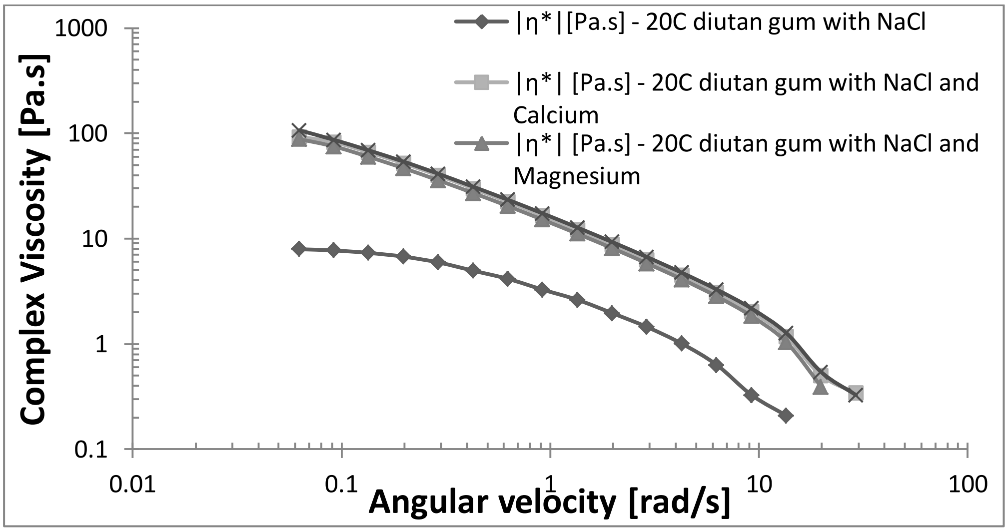

Fig. 20 Complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm) at 1 Pa and 20^0C

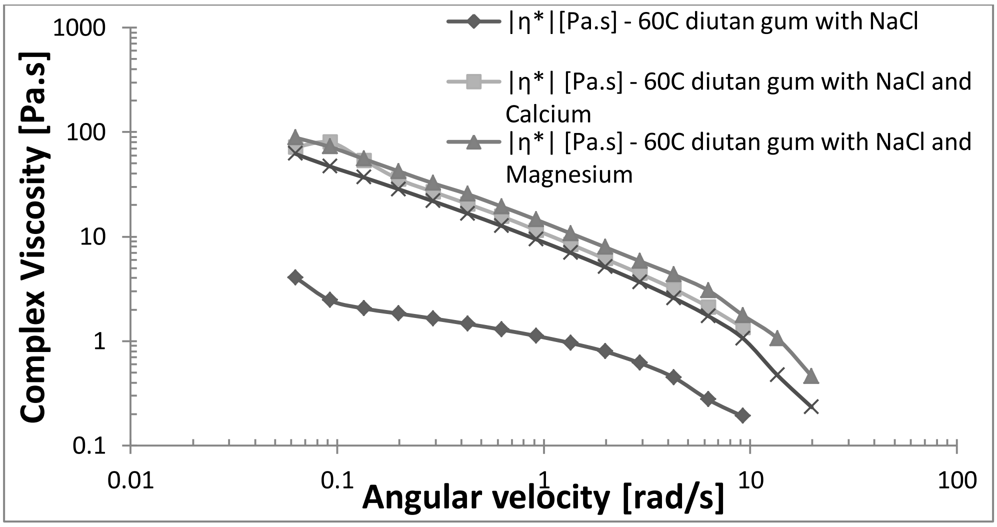

Fig. 21 Complex viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm) at 1 Pa and 60^0C

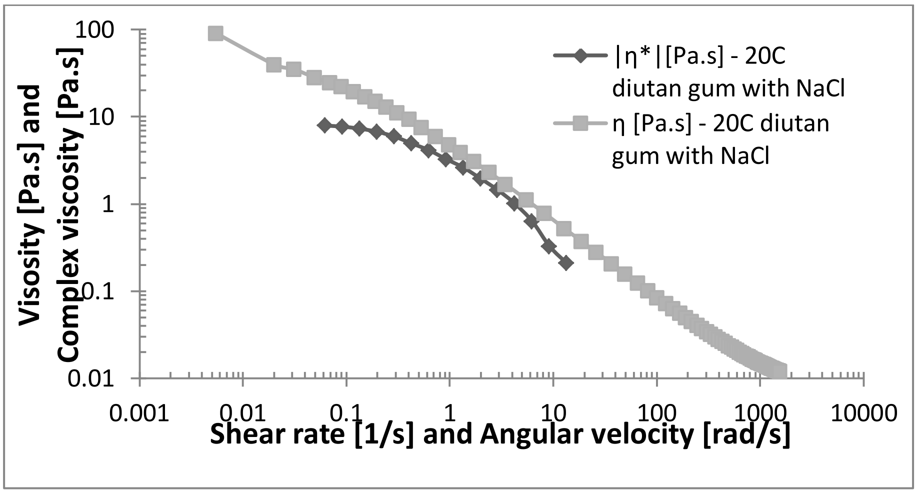

Fig. 22 Viscosity and Complex Viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 20^0C

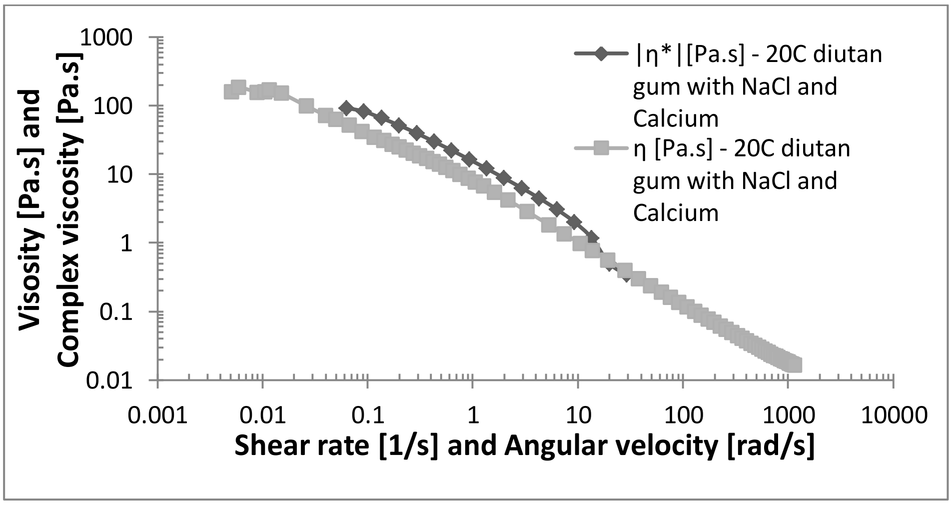

Fig. 23 Viscosity and Complex Viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) and Calcium (300 ppm) at 20^0C

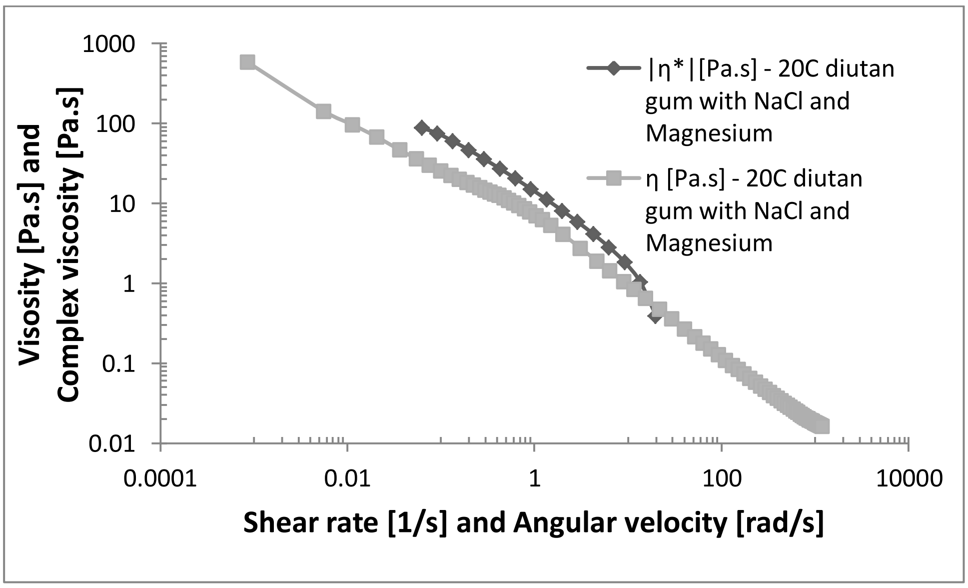

Fig. 24 Viscosity and Complex Viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) and Magnesium (300 ppm) at 20^0C

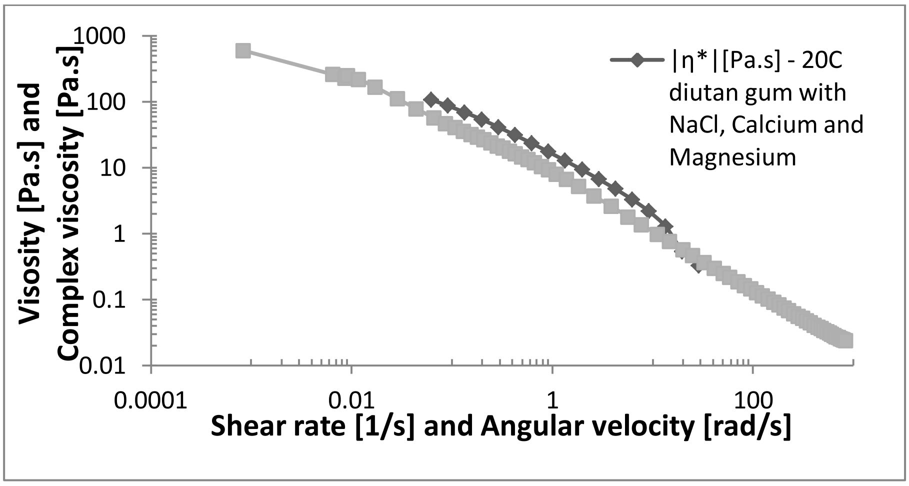

Fig. 25 Viscosity and Complex Viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm) at 20^0C

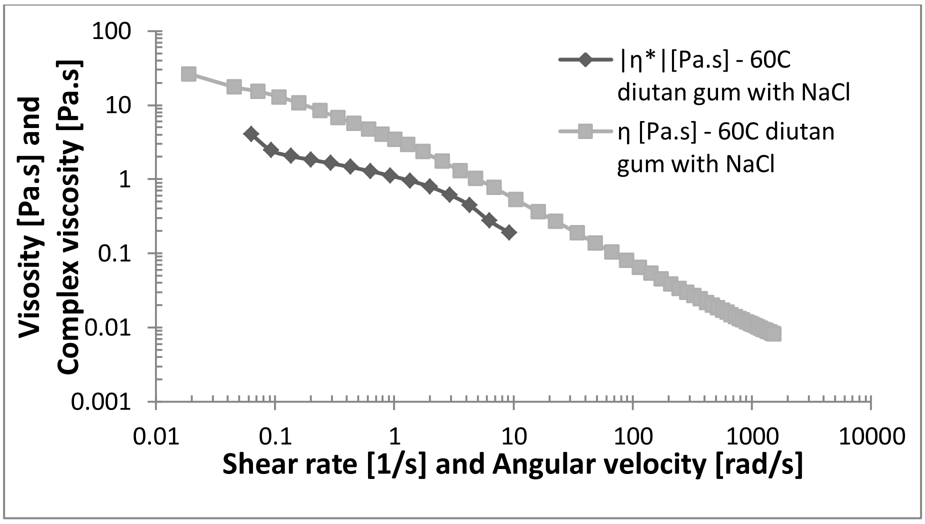

Fig. 26 Viscosity and Complex Viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) at 60^0C

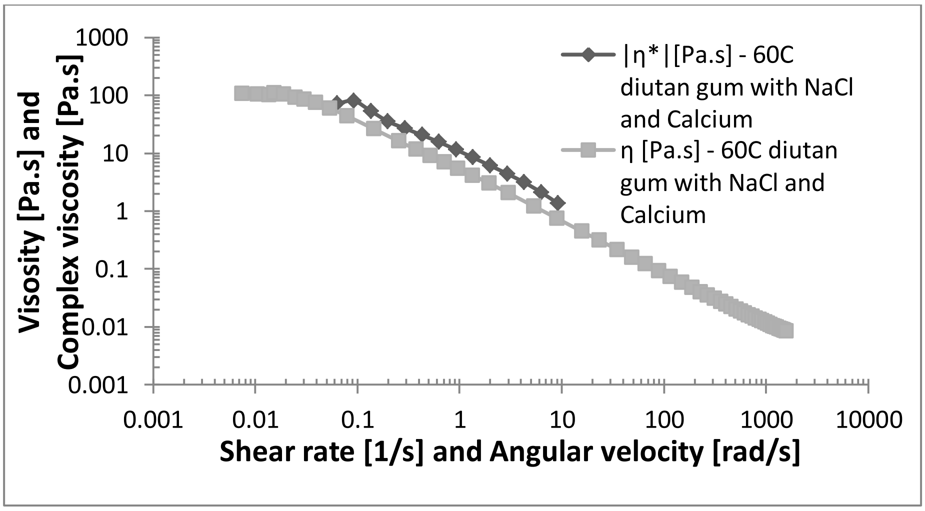

Fig. 27 Viscosity and Complex Viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) and Calcium (300 ppm) at 60^0C

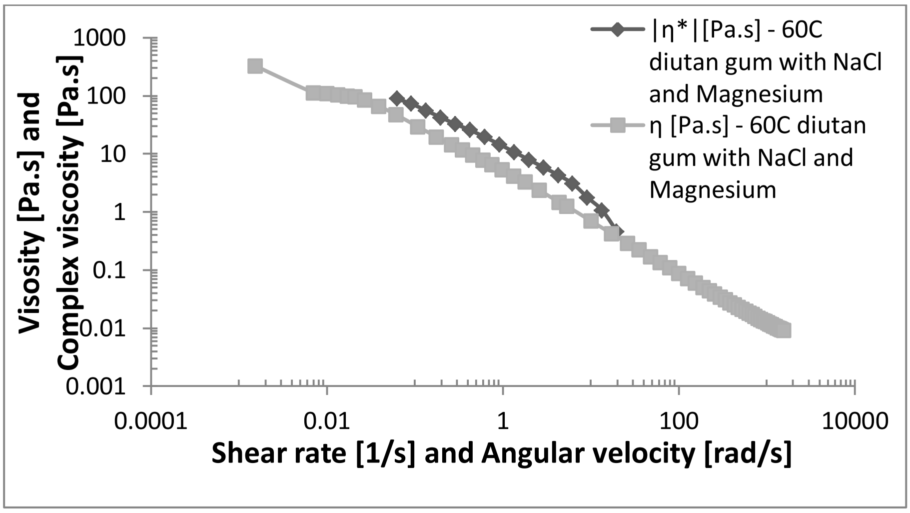

Fig. 28 Viscosity and Complex Viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm) and Magnesium (300 ppm) at 60^{0}C

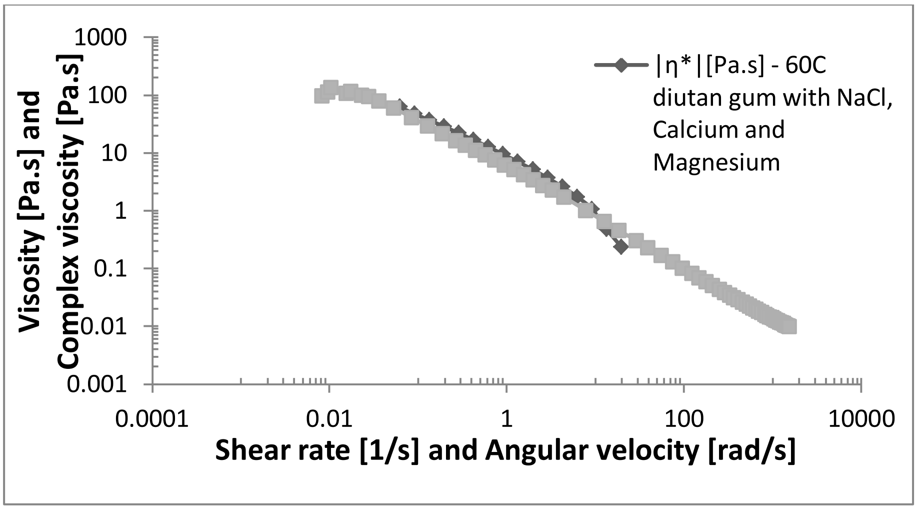

Fig. 29 Viscosity and Complex Viscosity of aqueous diutan gum solution (4300 ppm) with NaCl (40000 ppm), Calcium (300 ppm) and Magnesium (300 ppm) at 60^{0}C