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MODELING OF BURGER PARAMETERS FOR CMC-GUAR GUM BASED POLYMER NETWORK

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Abstract

Different gum concentrations of carboxymethyl cellulose (CMC)-guar gum (GG) mixtures (1.5, 2.0, 2.5%) and their volumetric mixing ratio (25:75, 50:50, 75:25) were analyzed for Burger model parameters by using response surface design approach based on creep-recovery measurements at 15, 25, 35 °C. Four component Burger model was used to characterize viscoelasticity of gum mixtures with experimental creep-recovery responses (J_0, J_1, t_1, η_0, Jr_0, Jr_1 and tr_1) and it was found to be satisfactory (R^2= 0.82-0.99) for the determination of the creep-recovery properties of gum mixtures. The high ratio of GG in concentrated CMC-GG mixture provided an increase in the elasticity in the strong or stiffer structure of gum mixtures at especially low temperatures when it was compared to CMC. However, at high temperature viscous property of the CMC-GG mixture was dominant. It was found that regressed parameters from Burger model were highly dependent to temperature with respect to both volumetric mixing ratio and concentration.

Keywords: Burger model, creep-recovery responses, CMC-GG mixture, viscoelasticity, rheology, response surface methodology

CMC-GUAR GAM POLİMER AĞI İÇİN BURGER MODELİNE AİT PARAMETRELERİN MODELLENMESİ

Öz

Karboksimetil selüloz (CMC)-guar gam (GG) karışımlarının sünme-geri dönüş ölçümlerine dayanan Burger model parametreleri farklı gam konsantrasyonları (%1.5, 2.0, 2.5) ve hacimsel gam karışım oranları (25:75, 50:50, 75:25), farklı sıcaklıklarda (15, 25, 35 °C) yanıt yüzey yaklaşımı kullanılarak analiz edilmiştir. Dört bileşenli burger model ve deneysel sünme-geri dönüş özelliklerinden yaralanarak (J_0, J_1, t_1, η_0, Jr_0, Jr_1 ve tr_1) gam karışımlarının viskoelastikiyeti karakterize edilmiştir ve gam karışımlarının sünme-geri dönüş özelliklerinin belirlenmesinde bu model (R^2= 0.82-0.99) tatmin edici bulunmuştur. Özellikle düşük sıcaklıklarda, yüksek GG içeren konsantre gam karışımları CMC ile karşılaştırıldığı zaman karışımın elastisitesini ve mukavemetini arttırmıştır. Öte yandan, yüksek sıcaklıkta karışımın viskoz özelliği baskındır. Burger model regresyon parametrelerinin hacimsel CMC-GG karışım oranı ve konsantrasyonuna kıyasla daha çok sıcaklığa bağlı olduğu bulunmuştur.

Anahtar kelimeler: Burger modeli, sünme-geri dönüş yanıtları, CMC-GG karışım, viskoelastikiyet, reoloji, yanıt yüzey yöntemi

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INTRODUCTION

Gums are the main ingredients in foods as a thickening and gelling agents to control viscosity (1). The use of two or more gums in a mixture is a common practice in the food industry due to synergistic interaction between or among themselves (1). Guar gum (GG) is used as an economical thickening and stabilizing agents in the food industry and is often combined with xanthan gum (XG), locust bean gum (LBG), or carboxymethyl cellulose (CMC) to increase synergistic changes in viscosity or gelling behaviour of complex food stucture (2).

When gums- either individually or in mixture- were used viscosity of that particular food need to be controlled as the rheological characterization of a mixed gums is important for product quality and shelf life. Due to viscoelastic nature of gums, elastic and viscous properties of gum solutions can be measured using small amplitude oscillatory shear (SAOS) and creep recovery tests as SAOS test gives the knowledge of dynamic properties such as elastic modulus (*G'*) and loss modulus (*G"*) in the linear viscoelastic region range (3). Creep-recovery tests are the most commonly used methods to define viscoelastic properties of materials (3). Creep and recovery are coupled test that creep test is obtained by applying a constant shear stress to the material, and deformation (strain) is recorded over the creep time. In the recovery part, shear stress is removed and again deformation is recorded over recovery time. The deformation supplies the alteration in molecular structure of material during this test. Burger model which is combined with Maxwell (4) and Kelvin-Voigt Model (5) connected in series determines the stress relaxation characteristics of viscoelastic samples from the resulting creep and recovery rheological measurement. This model is a mechanistic approach of viscoelasticity to simulate the linear relaxation of real viscoelastic materials at the small material strains. Each regressed coefficients (J_0, J_1, t_1, η_0, Jr_0, Jr_1 and tr_1) of Burger model signify to distinct viscoelastic characterization of mixed gums.

However, there is a limited number of studies on Burger model application into complex food or non-food systems. For example, Chompoorat et al. (6) analyzed diacetyl tartaric acid ester of monoglycerides (DATEM), ascorbic acid (AA), urea, and dithiothreitol (DTT) on viscoelastic properties of gluten using Burgers model. It was found that Burger model coefficients served as a tool to explain changing viscoelastic nature of gluten mixtures. On the other hand, Dogan et al. (7) used Burger model to find optimum gum mixtures which provides the highest resistance to deformation on pudding samples. Another study of Dogan et al. (8) is also related to the Burger model simulation on viscoelastic properties of ice cream samples as a function of different xanthan gum concentrations. Common side of these studies is the applicability of Burger model into complex food structured system using mechanical approach. From regressed Burger coefficients, it is possible to control physical characteristics related to the improvement of product design and quality. But, to our best knowledge there is no prior study on CMC-GG mixed gums based on creep-recovery parameters of the gum combination using Burger model.

Prediction of deformation knowledge of mixed gums with respect to different working conditions helps us to design and develop of new food materials for food industry. The aim of this study is to investigate effects of concentration of CMC-GG solutions, temperature and volumetric mixing ratio (CMC/GG) and their interaction terms on the creep-recovery parameters of CMC-GG mixture samples by modeling their viscoelastic behavior using Burger Model and to determine the relation between the regressed coefficients at the different design levels.

MATERIALS AND METHODS

Materials

Polymer powders of guar gum (GG) and sodium carboxylmethyl cellulose (CMC) (Sigma-Aldrich Corp, St Louis, MO), with nominal molecular weight of 700,000 g/mol were kindly provided by Dr. Kerim YAPICI, Department of Nanotechnology Engineering, Cumhuriyet University, Sivas, Turkey.

Preparation of gum solutions

The CMC and GG powders were dissolved in distilled water separately at 25 °C for 6 h using a magnetic stirrer with gentle shaking in order to prepare 1.5, 2 and 2.5% (w/v) stock solutions of CMC and GG. The CMC stock solution was thoroughly mixed operated at 150 rpm with GG solution at different volumetric mixing ratios of CMC:GG, such as 75:25; 50:50 and 25:75, respectively.

Measurements of rheological properties

Creep and recovery tests of the samples measurements were performed by using a stress controlled rheometer (Malvern Kinexus Pro, UK) fitted by a cone-and-plate system (50 mm diameter, 1° cone angle). A peltier plate assembly was used for temperature controlling purpose during the measurements with ±0.1 °C precision. Strain amplitude sweep over a stress was carried out under constant frequency to determine linear viscoelastic region storage. Elastic modulus (*G'*) and loss modulus (*G"*) of each sample changing with respect to oscillatory stress at a constant frequency of 1 Hz are given in Figure 1.

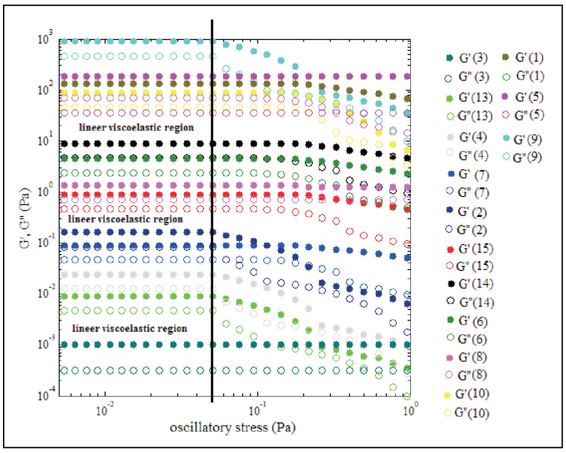

Figure 1. Variation of the loss (*G"*) and storage (*G'*) modulus with oscillatory stress at 1 Hz for all design samples

In the figure all curves show common viscoelastic region up to 0.05 Pa characterized by a constant modulus with respect to stress. So, creep- recovery tests were carried out under the constant shear stress of 0.05 Pa (in linear viscoelastic region) at three different temperatures (15, 25, and 35 °C) to measure the deformation of mixtures. All experiments were conducted in duplicate.

Creep-recovery tests include two parts, one of which is creep phase in which 0.05 Pa constant stress was applied for 120 s and the compliance values were recorded as a function of time. In the second part, which is recovery part, the applied stress was removed and then compliance values were also obtained as a func-tion of time for 120 s. In creep-recovery test, the stress response of samples under constant stress in a total time of 240 s was measured. Compliance value ($Jc(t)$, Pa^{-1}) shows deformation of mixed gums per unit stress as function of time (9) (data not shown).

Burger model

The effect of concentration of CMC-GG solutions, temperature and volumetric mixing ratio (CMC/GG) on the creep-recovery characteristics data of CMC-GG mixed gel were fitted into Burger model (10). Burger model has been commonly applied to study viscoelastic behavior of soft matter (11). Deformation behavior of material also was described by using Burger model coefficients. These regressed parameters from the model are given with the near corresponding compliance values depicted in Figure 2.

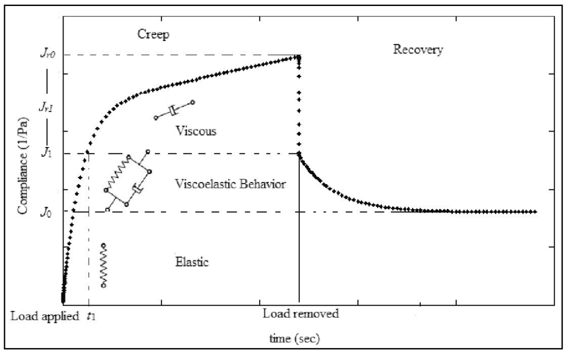

Figure 2. General representation of Burger creep and recovery model

Burger model, a combination of Maxwell and Kelvin-Voigt model, is represented by a spring and a dashpot; while, a parallel arrangement between spring and dashpot is used in Kelvin-Voigt model. It has two constitutive equations for creep and recovery parts. Equation (1) shows the model during creep:

$$Jc(t) = J_0 + J_1\left(1 - \exp\left(\frac{-t}{t_1}\right)\right) + \frac{t}{\eta_0} \qquad \text{Equation (1)}$$

where, $Jc\ (t)$ is creep compliance as function of time (t). The first element of Burger model is instantaneous shear compliance (J_0) corresponding to a spring or elastic modulus. The second element is delayed or retarded viscoelasticity (J_1). Retardation time (t_1) is a time of delayed elastic deformation. The last element is related to the zero shear rate viscosity of gum mixture (η_0). This element corresponds to an increase in deformation of dashpot. Burger model also yields recovery characteristics of gum mixtures. Equation (2) shows the Burger model during recovery:

$$Jr(t) = J_{r0} + J_{r1}\,(1 - \exp(\frac{-t}{t_{r1}})) \qquad \text{Equation (2)}$$

Equation (2) contained only 3 elements because there is no dashpot (pure viscous) during recovery phase. J_{r0} is the first part of recovery, corresponds to the spring of the Maxwell element. J_{r1} is the second recovery due to the Kelvin–Voigt element. t_{r1} represents the time that takes the gum mixture recovery step required for the elastic recovery of gum mixture.

Experimental design and statistical analysis

Response surface design was used to observe the effect of GG and CMC mixed gum on the parameters of the sample obtained from creep and recovery measurements. The relationship between the responses of J0, J1, t1, _0, Jr0, Jr1 and tr1 values and independent variables of temperature, concentration of CMC-GG solutions and volumetric mixing ratio (CMC/GG) were examined by using the Box-Behnken Design (BBD) with a quadratic model. A second order polynomial model was fitted to represent linear, interaction and quadratic effects of variables on Burger parameters. The experimental design of Box-Behnken model is represented in Table 1. A, B and C are values of temperature, concentration of CMC-GG solutions and volumetric mixing ratio (CMC/GG), respectively. The analysis was also performed in duplicates. The analysis of variance (ANOVA) and regression analysis were performed to define the coefficients of the predictive model and significant terms using MINITAB 16.0 (Minitab Inc. State College, PA, USA). The coefficient of determination (R2) was determined to check for the linearity between the predicted vs. experimental Burger parameters. Statistical analyses were accomplished using MINITAB 16.0 to test the significance of different rheological properties of mixed gums. The pairwise comparisons were made by Tukey's test with a significance level of 0.05. Pareto charts were plotted to represent the standardized effects of concentration of CMC-GG solutions, temperature and volumetric mixing ratio (CMC/GG) (via T values) on Burger model parameters such as J0, J1, t1, _0, Jr0, Jr1 and tr1 values.

RESULTS AND DISCUSSION

By calculating creep and recovery parameters of Burger model, the information of internal structure of gum mixtures at a given volumetric mixing ratio and temperature levels was obtained. All related model parameters were illustrated in Table 2 and Table 3.

Table 1. Experimental design for Burger model parameters using Box-Behnken response surface method (RSM)

Sample No.	Temperature (°C) (A)	Concentration of CMC-GG solutions (%) (B)	Volumetric mixing ratio (CMC/GG) (C)
1	35	1.5	50:50
2	25	2.0	50:50
3	15	1.5	50:50
4	15	2.0	25:75
5	35	2.0	25:75
6	25	2.5	75:25
7	15	2.5	50:50
8	25	1.5	25:75
9	35	2.5	50:50
10	35	2.0	75:25
11	25	2.0	50:50
12	25	2.0	50:50
13	15	2.0	75:25
14	25	2.5	25:75
15	25	1.5	75:25

Table 2. Creep model parameters of the mixed gum obtained from fitting of Eq.2*

Sample No.	J_0 (Pa^{-1})	J_1 (Pa^{-1})	t_1 (s)	η_0 (Pa.s)	R^2
1	0.020±0.002[e]	2.899±0.011[cd]	8.253±0.141[bcde]	83.230±4.240[f]	0.988±0.001
2	0.044±0.003[ab]	1.605±0.010[efg]	7.605±0.735[de]	192.690±10.670[e]	0.993±0.001
3	0.020±0.001[e]	0.909±0.025[g]	5.085±0.001[f]	249.550±0.960[b]	0.993±0.002
4	0.030±0.002[d]	1.916±0.014[ef]	8.106±0.030[bcde]	258.450±2.360[ab]	0.988±0.004
5	0.035±0.001[cd]	3.143±0.222[bcd]	10.951±0.233[a]	63.180±5.580[g]	0.982±0.006
6	0.047±0.001[a]	3.879±0.397[ab]	9.560±0.671[abc]	211.340±2.980[cd]	0.994±0.000
7	0.029±0.000[d]	2.518±0.211[de]	8.205±0.113[bcde]	271.750±1.980[a]	0.996±0.001
8	0.043±0.005[ab]	3.705±0.120[abc]	9.105±0.028[abcd]	214.330±2.970[cd]	0.995±0.001
9	0.032±0.000[cd]	4.324±0.490[a]	10.195±0.098[a]	62.190±2.730[g]	0.994±0.003
10	0.028±0.000[d]	3.069±0.080[bcd]	9.492±0.212[abcd]	73.070±3.540[fg]	0.991±0.005
11	0.038±0.002[bc]	1.455±0.067[fg]	7.700±0.071[cde]	200.650±0.760[de]	0.989±0.005
12	0.046±0.000[a]	1.613±0.021[efg]	7.105±0.028[e]	203.730±4.950[cde]	0.987±0.008
13	0.029±0.000[d]	1.618±0.071[efg]	9.099±0.040[abcd]	266.730±2.120[a]	0.984±0.018
14	0.045±0.001[a]	4.196±0.006[a]	9.936±1.203[ab]	218.790±2.040[c]	0.992±0.001
15	0.048±0.000[a]	3.637±0.534[abc]	9.713±0.888[ab]	202.890±0.930[cde]	0.993±0.002

* Values followed by the same letter in a column are statistically different ($p<0.05$)

Table 3. Recovery model parameters of the mixed gum obtained from fitting of Eq.3*

Sample No.	Jr_0 (Pa^{-1})	Jr_1 (Pa-1)	tr_1 (s)	R^2
1	56.790±0.620[c]	55.790±0.630[c]	39.030±0.550[a]	0.989±0.003
2	248.890±0.250[b]	248.070±0.080[b]	21.602±0.790[b]	0.993±0.002
3	357.060±0.100[a]	356.680±0.420[a]	18.080±0.000[b]	0.993±0.002
4	357.400±0.580[a]	356.200±0.030[a]	19.090±0.020[b]	0.988±0.004
5	56.890±1.170[c]	55.740±0.860[c]	39.900±1.320[a]	0.991±0.007
6	249.610±0.680[b]	249.030±0.020[b]	20.280±0.000[b]	0.994±0.000
7	356.860±0.070[a]	356.300±0.060[a]	21.090±0.390[b]	0.996±0.001
8	249.140±0.070[b]	248.050±0.040[b]	19.610±0.330[b]	0.995±0.001
9	58.180±0.070[c]	55.180±0.080[c]	40.230±0.803[a]	0.994±0.003
10	56.440±0.070[c]	55.570±0.350[c]	37.180±2.820[a]	0.996±0.002
11	249.604±0.020[b]	248.570±0.780[b]	21.090±0.050[b]	0.989±0.005
12	249.002±0.060[b]	248.040±0.000[b]	21.470±0.540[b]	0.992±0.001
13	357.205±0.180[a]	356.530±0.480[a]	18.480±0.340[b]	0.992±0.006
14	253.420±6.120[b]	252.190±5.880[b]	19.080±0.140[b]	0.992±0.001
15	249.510±0.450[b]	248.530±0.750[b]	21.050±0.330[b]	0.993±0.002

** Values followed by the same letter in a column are statistically different ($p<0.05$)

It was found that J_0 values were smaller than J_1 leading to higher viscoelastic behavior than elastic nature of gum mixtures. At the maximum concentration of CMC and GG solutions level (2.5 %), J_1 values were higher due to intermolecular interaction between GG-CMC mixture depended strongly on the concentration of CMC and GG solutions. The interactive effects of significant variables were represented in contour plots as shown in Figure 3.

The maximum response is referred to a surface confined in the smallest ellipse in a contour plot. The perfect interaction between the independent variables can be shown when elliptical contours are obtained (11). Figure 3a shows that the effect of concentration of CMC and GG solutions and temperature on J_1 with the interactive effect of concentration of CMC and GG solutions and positive significant effect of temperature (Figure 3a). As shown in Figure 3a, the viscoelastic response, J_1, for high volumetric mixing ratio of GG was significantly increased as the temperature increased. Especially, J_1 reached maximum value of 4.19 Pa^{-1} (25 °C) at high concentration of CMC and GG solutions (2.5%) and GG ratio (25:75) in the mixture (Table 2). Higher content of GG caused an increase in the viscoelasticity by 7.55% when compared to CMC (Table 2, Samples 6 and 14). Moreover, increased temperature also provided an increasing effect on the viscoelasticity (Table 2, Samples 4 and 5). For creep phase parameter of J_1, the increase in the ratio of GG reinforced the viscoelastic structure of mixed gum by decreasing the viscosity especially at high concentration of 2.5(%) at 35 °C as in Figure 3a. At 35 °C, maximum viscoelasticity was also observed with high mixing concentration ratio of 50:50 (Table 2, Sample 9). Hence, higher permanent deformation of mixed gum was obtained at high values of J_1.

When highly extend of GG in mixture, the viscoelasticity of gum mixture can be attributed to entanglement network structure due to more elastic nature of GG than that of CMC. On the other hand, retardation time, t_1 was directly related to viscoelastic properties which was similar to J_1. So, it has showed similar trends as in J_1. Increasing concentration of CMC and GG solution did not significantly change t_1 (P>0.05), but it was highly dependent on the mixed gum ratio. η_0 gives the degree of zero shear rate viscosity of gum mixtures and was significantly changed with temperature. η_0 values ranged at 62.19-266.73 (Pa.s) (Table 2). It was noted that η_0 values decreased with increasing temperature (Sample 5 and 9, Table 2). A significant synergetic effect of CMC-GG mixture was also observed even at low concentration (1.5%) and the ratio of 50:50 mixture. Thus, the viscous behavior of CMC-GG solutions also increased up to 83 Pa.s at 35 °C (Table 2). However, it was found that gum mixtures showed higher η_0 values which were expressed more stiff structure of gum mixture at 15 °C.

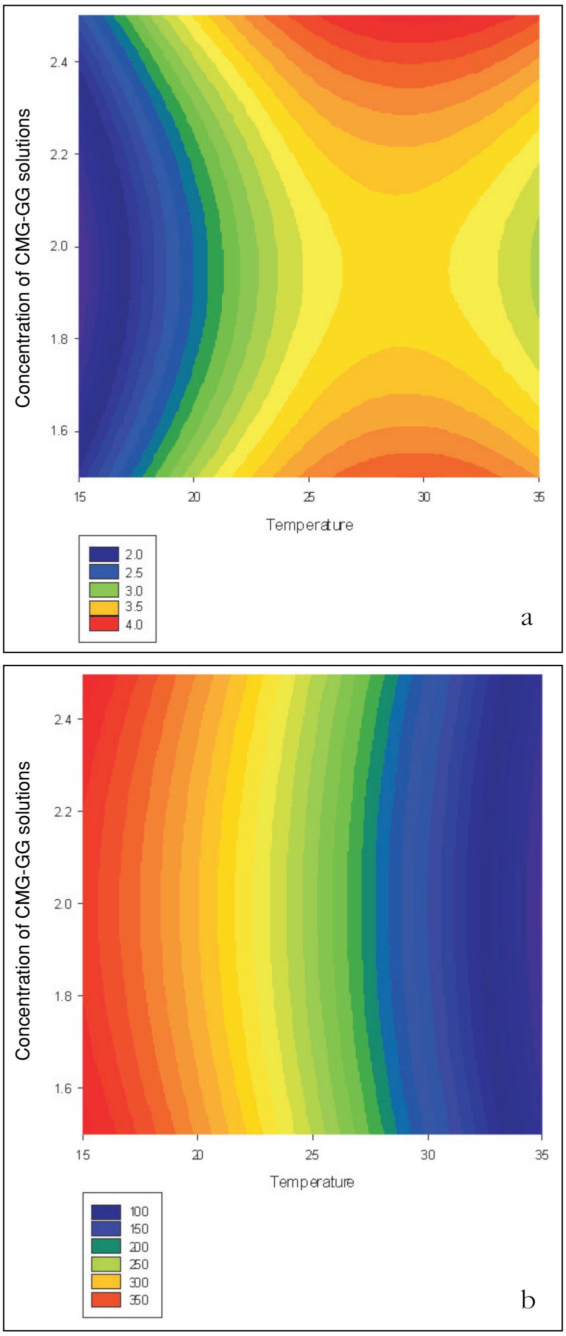

Figure 3. Contour plots showing interactive effect of concentration of CMC-GG solutions and temperature for a) J_1, b) Jr_1 at constant volumetric mixing ratio gum ratio (25:75).

In recovery phase, Jr_0 is related to the elastic nature of gum mixture, whereas Jr_1 is related to the viscoelastic behavior of concentration of CMC and GG solutions (Table 3). These parameters showed the same trend in which their values considerably changed with temperature. Effect of CMC and GG solutions's concentration and temperature on Jr_1 represented in Figure 3. Increasing temperature, recovery coefficients remarkably decreased due to viscous deformation of gum mixtures and irreversibly deformation of elastic chains between CMC-GG molecules. Also, tr_1 values reached to higher values at 35 °C (Table 3). On the other hand, at low temperature, recovery was obtained at a shorter time after the applied deformation, and gum mixtures became more stiff structure. For example, at 15 °C, viscoelastic recovery coefficients amplified to the order of 350 Pa^{-1} due to giving quick response of gum chains compared to 35 °C when the applied force removed at high volumetric mixing (CMC/GG) ratio in the mixture.

After the determination of the Burger model parameters of the CMC-GG mixed gum, Box-Benhken design was applied to model as a function of temperature, concentration of CMC and GG solutions, and volumetric mixing ratio (CMC/GG). Table 4 represents the established predicted models, constants, coefficient of determination values (R^2) and lack of fit values. R^2 values of the creep model parameters for J_0, J_1, t_1 and η_0 values were in the range of 0.82-0.99, whereas R^2 values of the recovered model parameters for Jr_0, Jr_1 and tr_1 values were in the range of 0.98-0.99. There was also insignificant lack of fit for all models. The degree of efficacy of varying process conditions on the burger parameters can be deduced by comparing the magnitude of the coefficients of the second order model (Table 4). Temperature is the most important factor with the highest coefficient 0.016, 0.0809, 1.049, 95.59, 150.01, 150.43 and 9.95 for J_0, J_1, t_1, η_0, Jr_0, Jr_1 and tr_1 values, respectively (Table 4).

The standardized effect of the independent variables (concentration of CMC and GG solutions, temperature and volumetric mixing ratio (CMC/GG), quadratic effects and their interactions on Burger model parameters such as J_0, J_1, t_1, η_0, Jr_0, Jr_1 and tr_1 values was also visualized by Pareto chart (Figures 4a-g), in which the process variables below the vertical line are considered insignificant and those above the line with a negative sign also implied significant effect but in opposite mode. Thus, only temperature and gum concentration positively affected J_1, t_1 and tr_1, whereas only gum concentration had a positive effect on J_0 (Figures 4a-g).

CONCLUSIONS

Creep and recovery Burger model parameters of CMC-GG gum mixtures were analyzed with respect to temperature, concentration of CMC and GG solutions, and volumetric mixing ratio (CMC/GG). CMC-GG mixtures were also modeled in order to characterize distinct elastic, viscoelastic and viscous behavior of gum mixture using response surface design method in terms of Burger model parameters. The model was confirmed that parameters from Burgers model could assist in explaining changes in rheological structures. All elastic coefficients were found smaller order than viscoelastic coefficients. Viscoelastic characteristics of gum mixture were more pronounced at higher ratio of GG indicating a higher regressed viscoelastic coefficients of GG than CMC especially in concentrated gum mixture. On the other hand, recovery coefficients were found highly temperature dependent due to higher contribution of viscous deformation of gum mixtures and irreversibly deformed elastic chains between GG-CMC molecules at high temperature.

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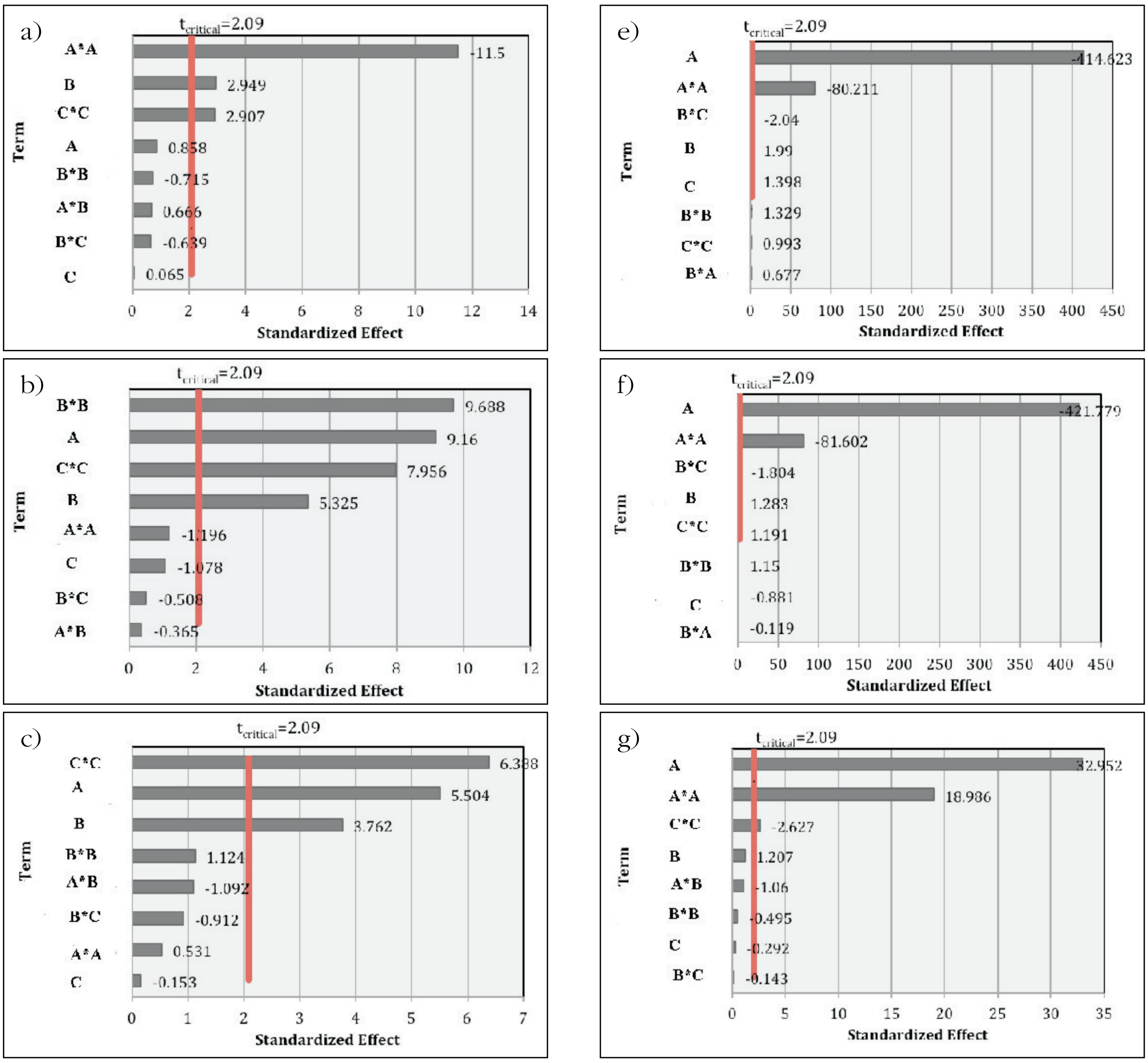
Table 4. Predicted models, R^2 values and lack of fit values for the parameters of CMC-GG mixed gums

Parameter	Predicted models	R^2	R^2_{adj}	R^2_{pred}	*Lack of fit*
J_0	$J_0=0.043-0.016A^2+0.004C^2+0.003B$	0.89	0.85	0.76	0.89^{ns}
J_1	$J_1=1.558+1.260B^2+1.035C^2+0.809A+0.471B$	0.93	0.89	0.79	0.27^{ns}
t_1	$t_1=7.469+1.793C^2+1.049A+0.717B$	0.82	0.77	0.70	0.16^{ns}
η_0	$\eta_0=199.02-39.390A^2+7.060B^2+5.760C^2-95.590A-10.810AB$	0.99	0.99	0.99	0.34^{ns}
Jr_0	$Jr_0=249.180-42.720A^2-150.010A$	0.99	0.99	0.99	0.09^{ns}
Jr_1	$Jr_1=248.220-42.840A^2-150.430A$	0.99	0.99	0.99	0.11^{ns}
tr_1	$tr_1=21.390+8.440A^2-1.170C^2+9.950A$	0.98	0.98	0.97	0.10^{ns}

ns: Not significant, A: Temperature, B: Concentration of CMC-GG solutions, C: Volumetric gum ratio



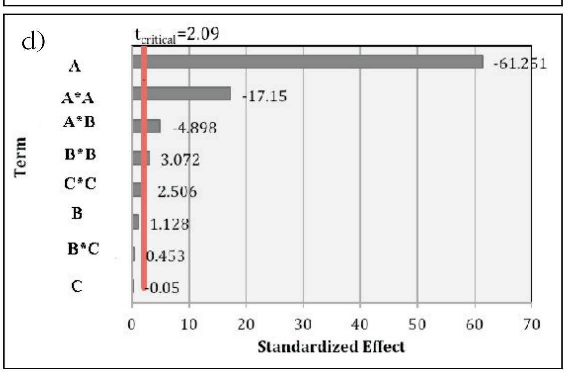


Figure 4. Pareto charts showing the standardized effects of temperature (A), concentration of CMC-GG solutions (B), and volumetric mixing ratio (C) (via T values) on Burger model parameters such as J_0, J_1, t_1, η_0, Jr_0, Jr_1 and tr_1 values.

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