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RHEOLOGICAL PARAMETERS OF XANTHAN GUM / PECTIN SOLUTIONS AS A FUNCTION OF TEMPERATURE AND COMPOSITION

PARÁMETROS REOLÓGICOS DE SOLUCIONES DE GOMA XANTANA / PECTINA EN FUNCIÓN DE LA TEMPERATURA Y COMPOSICIÓN

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Abstract

The rheological behavior of individual solutions and blends of xanthan gum (0.1 to 0.5 %) and pectin (1.6 to 2.0 %) was studied at 25, 50 and 75 °C using a D-optimal mixture experimental design. Pectin and xanthan gum solutions were found to be Newtonian and non-Newtonian fluids, respectively. The effect of the temperature on the viscosity of pectin solutions and the consistency index (K) of xanthan gum were modeled by the Arrhenius equation. The xanthan gum/pectin mixtures predominantly behaved as pseudoplastic fluids and were described by the power law model. Mathematical models were developed to predict the flow behavior index and the consistency index as a function of the blends composition, at the studied temperature. The activation energy of flow was expressed in terms of the concentrations of the components of the mixtures. The obtained mathematical models are useful for predicting the rheological parameters of pectin and xanthan gums blends and for evaluating possible applications.

Keywords: xanthan gum, pectin, xanthan gum/pectin mixtures, flow behavior index, consistency index, activation energy.

Resumen

Se estudió el comportamiento reológico de soluciones individuales y mezclas de goma xantana (0.1-0.5 %) y pectina (1.6-2.0 %) a 25, 50 y 75 °C, mediante un diseño experimental de mezclas D-óptimo. Las soluciones de pectina y de goma de xantana mostraron un comportamiento newtoniano y no newtoniano, respectivamente. El efecto de la temperatura sobre la viscosidad de soluciones de pectina y el índice de consistencia (K) de las soluciones de goma xantana se modeló mediante la ecuación de Arrhenius. Las mezclas de goma xantana/pectina se comportaron predominantemente como fluidos pseudoplásticos y se describieron por el modelo de la ley de la potencia. El índice de comportamiento de flujo y el índice de consistencia se modelaron en función de la composición de las mezclas, a las temperaturas estudiadas. La energía de activación de flujo se expresó en función de la concentración de los componentes de la mezcla. Los modelos matemáticos obtenidos son de utilidad para predecir los parámetros reológicos de mezclas de pectina y goma xantana y para evaluar posibles aplicaciones.

Palabras clave: goma xantana, pectina, mezclas de goma xantana/pectina, índice de comportamiento de flujo, índice de consistencia, energía de activación.

1 Introduction

The extensive use of hydrocolloids, highly hydrophilic polymers, is partially due to their capacity to modify the rheological properties of aqueous food systems (Saha and Bhattacharya, 2010; Li and Nie 2016; Juszczak *et al*., 2004).

Along with ionic strength, pH, electrical charge, degree of dispersion, molecular weight, chemical structure, and methods of preparation, the temperature and composition affect the rheological characteristics of the hydrocolloids (Casas *et al*., 2000; Casas-Alencáster and Pardo-García, 2005). The composition, concentration, type and nature of the

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hydrocolloids are the first variables to be considered for preparing a solution with specific rheological properties (Casas *et al*., 2000).

Some hydrocolloids like arabic gum, give low viscosities at relatively high concentrations (30 %), but a similar range of viscosities can be obtained with carboxymethylcellulose or xanthan gum at low concentrations (~1 %) (Sanchez *et al*., 2002). On the other hand, highly viscous dispersions can be formed with guar gum (6000-7500 mPas), locus bean gum (3500 mPas), gum tragacanth (4000 mPas), at only 1 % solids (Sittikijyothin *et al*., 2005).

It has been observed that the combination of hydrocolloids can lead to enhanced viscosity of the solutions (Saha and Bhattacharya, 2010). For instance, xanthan gum/guar gum and xanthan gum/locus bean mixtures solutions showed a higher combined viscosity (> 3200 mPa; > 1000 mPa) than the viscosity of their individual solutions, at 25 °C (Casas *et al*., 2000). A synergistic interaction has also been reported for hydroxypropyl guar gum/carboxymethyl cellulose and guar gum/pectin blends (Zhang and Kong, 2006; Satyamaiah *et al*., 2014).

The rheological characterization of different hydrocolloid blends is a fundamental part in the development of new food formulations and products. This study is focused on determining the rheological parameters of xanthan gum and pectin solutions and their blends as a function of concentration and temperature.

2 Materials and methods

2.1 *Raw material and mixtures preparation*

Citrus pectin with 69 % of esterification (DEQ, Mexico), xanthan gum (G1253-Sigma-Aldrich, USA) and distilled water were used for experimentation.

A set of aqueous mixtures of pectin (1.6-2.0 %, w/w), and xanthan gum (0.1-0.5 %, w/w) were prepared, as detailed in Table 1, at 60 °C under constant stirring (350 rpm) on a heating plate (Cimarec, Thermo Scientific, USA). Then, solutions were homogenized, and air bubbles were removed by ultrasound at 35 kHz for 30 min (Ultrasonic bath VWR, China). Individual solutions of xanthan gum (0.1 and 0.5 %, w/w) and pectin (1.6 and 2.0 %, w/w) were prepared under the same conditions. The pH of the solutions of pectin, xanthan gum, and its mixtures was measured using a pH-Meter Orion 3 Star (Thermo Scientific, USA).

Table 1. Composition of the aqueous mixtures of pectin and xanthan gum

Mixture	C_{XG} (%)	C_P (%)	C_W (%)
1	0.10	2.00	97.90
2	0.50	1.60	97.90
3	0.43	1.93	97.63
4	0.50	2.00	97.50
5	0.10	2.00	97.90
6	0.37	1.87	97.77
7	0.30	2.00	97.70
8	0.50	2.00	97.50
9	0.30	1.80	97.90
10	0.30	1.80	97.90
11	0.50	1.80	97.70
12	0.33	1.93	97.73
13	0.50	1.80	97.70
14	0.50	1.60	97.90
15	0.23	1.93	97.83
16	0.43	1.73	97.83

C_{XG} - xanthan gum concentration, C_P - pectin concentration and C_W - water concentration. All concentrations were expressed in % (w/w)

2.2 *Experimental design*

A D-optimal experimental mixture design (Design-Expert 7, version 7.1.5) was used to define the concentration levels of the mixtures components (Table 1) and to carry out the statistical analysis of the results. A distinctive characteristic of the selected experimental design is that the mixture components cannot be changed independently since their sum must be equal to 100 %. This means that the mixture factors are expressed as a fraction of the total amount, and their experimental ranges are between 0 and 100 % (Mura *et al*., 2005).

2.3 *Rheological parameter measurements*

The rheological measurements of individual solutions of pectin and xanthan gum, and of their mixtures were performed in a rheometer (MCR XX1, Anton Paar, Austria), using a parallel plate device of 50 mm diameter (1 mm gap). A linear scale flow test was applied varying shear rate ($\dot{\gamma}$) from 0-300 s^{-1}, at 25, 50 and 75 °C. For each solution, the tests were run randomly and replicated three times. The power law model was used to describe the rheological properties of xanthan gum solutions and pectin-xanthan gum mixtures, Eq. (1) (Guerra-DellaValle *et al*., 2009). Solutions containing only pectin were analyzed using a Newtonian model, Eq. (2) (Wang *et al*., 2008; Marcotte *et al*., 2001a).

$$\tau = K\dot{\gamma}^n \tag{1}$$

$$\mu = \frac{\tau}{\dot{\gamma}} \tag{2}$$

where τ is the shear stress (Pa), K is the consistency index (Pa s^n), n is the flow behavior index (dimensionless), γ is the shear rate (s^{-1}) and μ is the viscosity (Pa s).

2.4 Activation energy of flow

The effect of temperature on the consistency index (K) was determined with the linearized Arrhenius equation, Eq. (3) (Ramírez-Arreola *et al.*, 2006).

$$\ln K = \ln A - \frac{E_a}{R}\frac{1}{T_a} \tag{3}$$

where K is the consistency index (Pa s^n), A is the pre-exponential factor (Pa s^n), Ea is the activation energy of flow (kJ mol^{-1}), R is the universal gas constant (8.314 J mol^{-1}K^{-1}) and T_a is the absolute temperature (K).

3 Results and discussion

3.1 Rheological properties of individual solutions of pectin and xanthan gum
Pectin aqueous solutions

Aqueous solutions containing 1.6 and 2.0 % of pectin had a pH of 3.04 ± 0.00, similar to pH= 2.80 ± 0.04 reported by Yaseen *et al.* (2005) for a pectin solution (concentration not specified). At the three studied temperatures, pectin solutions behaved as Newtonian fluids, the rheogram (Fig. 1) clearly shows that the shear stress is proportional to the shear rate. These results are consistent with those reported for citrus pectin solutions with concentrations of 1.0 and 2.0 % (Baümler *et al.*, 2013). On the contrary, it has been reported that citrus pectin solutions above 0.4 % (Methacanon *et al.*, 2014) and between 1 and 5 % (Marcotte *et al.*, 2001a), did not display Newtonian behavior. These contrasting results could be related to the molecular weight, degree of esterification and extraction conditions of the pectin (Sriamornsak, 2003). Larger number of interactions between pectin molecules with water results in a greater expansion of the molecules and the formation of large three-dimensional networks, which favors non-Newtonian behavior (Sato *et al.*, 2008).

As shown in Table 2, the viscosity of pectin solutions increased with rising concentration and decreased with increasing temperature. The effect of the concentration could be due to the intermolecular forces and strength of water-solute hydrogen bonds that restrict the molecular motion (Constenla *et al.*, 1989). When high pectin amount is dissolved in water, the viscosity rises because of the increment in hydrogen bonding with pectin hydroxyl groups (Azoubel *et al.*, 2005; Guimarães *et al.*, 2009). Moreover, the most significant change in viscosity was observed when the temperature increased from 25 to 50 °C. These changes could be attributed to a more pronounced variation in the intermolecular separation, caused by a greater thermal expansion (Guimarães *et al.*, 2009).

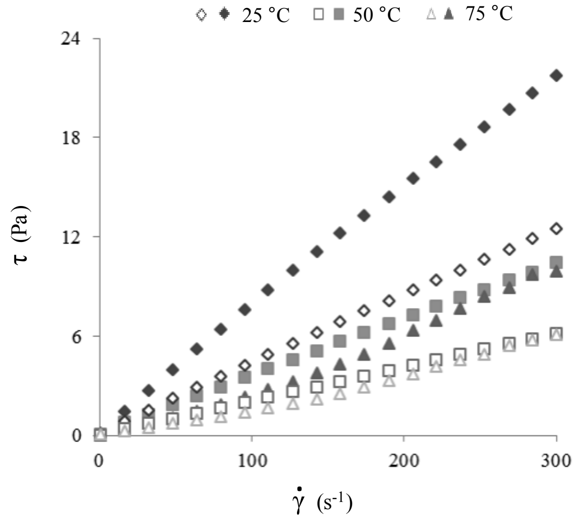
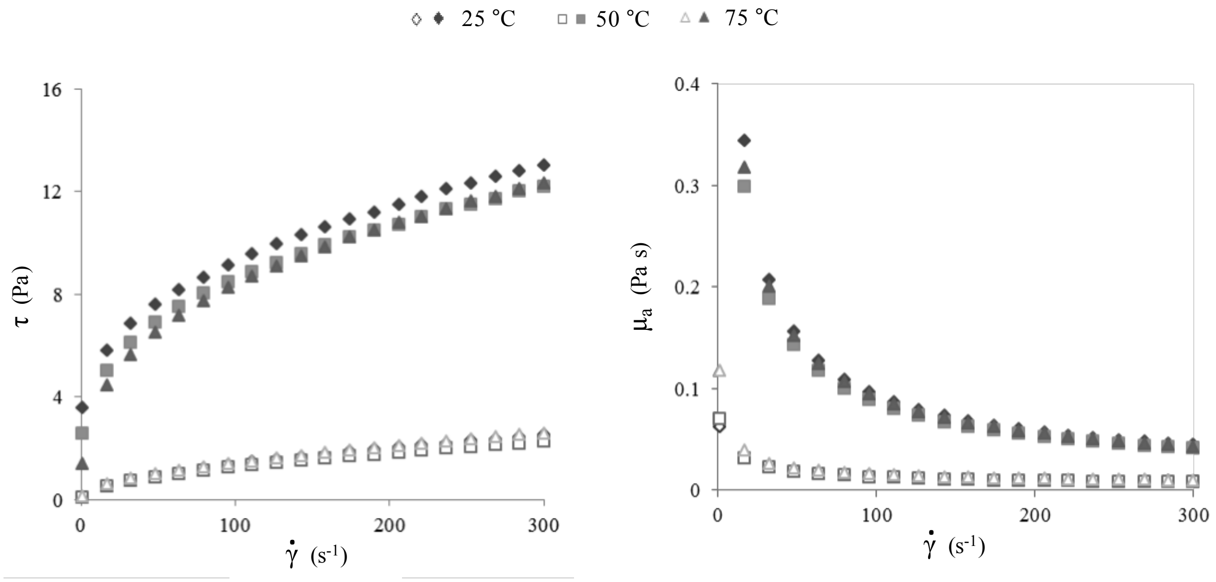


Fig. 1. Rheograms of aqueous solutions of pectin at different temperature and concentration (1.6 % - open symbols; 2.0 % - filled symbols). Shear stress (τ) and shear rate ($\dot{\gamma}$).

Table 2. Viscosity at different concentration and temperature, and activation energy of aqueous solutions of pectin

C_P (%,w/w)	μ^* (Pa s)			E_a (kJ mol^{-1})	R^2_{adj}
	25 °C	50 °C	75 °C		
1.6	0.041±0.00	0.020±0.00	0.019±0.00	13.71	0.834
2.0	0.071±0.00	0.034±0.00	0.033±0.00	13.68	0.815

C_P - pectin concentration, μ - viscosity, E_a- activation energy of flow, R^2_{adj} - adjusted coefficient of determination. *Mean and standard deviation of 3 values.



a)

b)

Fig. 2. Dependence of a) shear stress (τ) and b) apparent viscosity (μ_a) on shear rate ($\dot{\gamma}$) of aqueous solutions of xanthan gum at different temperature and concentration (0.1 % - open symbols; 0.5 % - filled symbols).

Table 3. Power-law parameters at different concentration and temperature and, activation energy of aqueous solutions of xanthan gum

	C_{XG} (%,w/w)	Temperature (°C)			E_a (kJ mol^{-1})	R^2_{adj}
		25	50	75		
n^*	0.1	0.52±0.00	0.57±0.01	0.63±0.00	-3.61	0.988
	0.5	0.24±0.00	0.27±0.00	0.38±0.00	-7.91	0.902
K^* (Pa s^n)	0.1	0.13±0.00	0.09±0.00	0.06±0.00	13.47	0.995
	0.5	3.17±0.00	2.57±0.09	1.43±0.04	13.71	0.906

C_{XG} - xanthan gum concentration, n - flow behavior index, K - consistency index, E_a - activation energy of flow, R^2_{adj} - adjusted coefficient of determination. *Mean and standard deviation of 3 values.

The temperature dependency of the pectin solutions viscosity was modeled by the Arrhenius equation (Table 2). The activation energy values (13.71, 13.68 kJ mol^{-1}) indicate a similar temperature sensitivity of the 1.6 and 2 % pectin solutions. High activation energies were reported in 1 and 3 % citrus pectin solutions (19.7 and 33.4 kJ mol^{-1}) and 1.4 % grapefruit pectin solution (15.77 kJ mol^{-1}) (Marcotte *et al.*, 2001a; Arslan and Toğru1, 1996). These discrepancies could be due to the type of pectin and particular experimental conditions (Lopes da Silva *et al.*, 1994).

3.2 Xanthan gum aqueous solutions

In the experimental temperature range, the pH of the 0.1 % and 0.5 % xanthan gum solutions was 6.58 ± 0.21 and 5.85 ± 0.04, respectively. For similar gum concentrations Yaseen *et al.* (2005) reported a pH= 4.24, at 4 °C.

Solutions of xanthan gum showed a rheology of pseudoplastic fluid (Fig. 2a); therefore, its apparent viscosity decreased with increasing shear rate from 0 to 300 s^{-1} (Fig. 2b) at the studied temperatures. Similar behavior was observed in 0.5 to 1.3 % xanthan gum solutions (Speers and Tung, 1986; Xuewu *et al.*, 1996).

The rheological behavior of xanthan gum solutions was modeled with the power law equation, Eq. (1). The flow behavior index (n) of the solutions decreased when the xanthan gum concentration passed from 0.1 to 0.5 % (Table 3). This reduction in the n values implies a tendency towards a pseudoplastic behavior of the xanthan gum solutions. Marcotte *et al.* (2001b) observed the same trend in more concentrate xanthan gum solution (2 %) at temperatures between 20 and 80 °C. The effect of xanthan gum concentration on pseudo plasticity could be attributed to the dissociation of the carbonium ions of the side chains of the gum and the formation of aggregates through numerous hydrogen bonds (Sworn, 2009).

Regarding the consistency index (K) of xanthan gum solutions (Table 3), it can be observed that it increased with concentration. A 0.5 % xanthan gum solution exhibited the highest K value (3.17 Pa s^n) at 25 ºC, which is four times lower than the reported for a 1 % xanthan gum solution (12.46 Pa s^n) at 20 °C (Tipvarakarnkoon and Senge 2008).

The relationship between the temperature and the rheological parameters (flow behavior index and the consistency index) of xanthan gum solutions were modeled by the Arrhenius equation ($R^2 > 0.900$), as shown in Table 3. The low activation energy values obtained for the flow behavior index indicate that the temperature has not influenced on this parameter. Meanwhile, the Ea was concentration dependent as previously reported by Speers and Tung (1986).

The sensitivity to the changes of temperature of both xanthan gum samples was of similar magnitude, according to the obtained activation energy values (E_a= 13.47 and 13.71 kJ mol^{-1}). The response of K to the rise of temperature from 25 to 75 °C could be associated with the transition from a five-fold helical structure, stabilized by intermolecular and intramolecular hydrogen bonds, to a random coil structure with a limited number of hydrogen bonds (Khouryieh *et al*., 2006).

3.3 Xanthan gum and pectin mixtures

In average, the pH of the 16 mixtures was 3.27 ± 0.09, which is between the pH values of the individual solutions of pectin (pH= 3.04) and xanthan gum (pH= 5.85 and 6.58). The pH values of the mixtures seem to be influenced by the greater proportion of pectin in the blends.

At the studied experimental conditions, the pectin-xanthan gum mixtures displayed a non-Newtonian character, which was found to be described by the power law. The obtained consistency index and flow behavior index were analyzed according to the D-optimal experimental mixture design (Design-Expert 7, version 7.1.5), to fit the appropriate model, to test it for goodness of fit (R^2) and to verify the adequacy of the regression model in terms of a lack-of-fit test through the ANOVA (not shown).

3.4 Statistical analysis of rheological parameters

Table 4 summarizes the obtained equations to predict the flow behavior index and the consistency index as a function of the mixtures composition, at 25, 50 and 75 °C. It can be seen that the flow behavior index varied linearly with the concentration of the components. As for the consistency index, it was also significantly affected by the concentration linear terms of xanthan gum and pectin, and the interaction $C_{XG} - C_P$ ($p < 0.05$).

Table 4. Predictive equations for flow behavior index and consistency index of mixtures of xanthan gum and pectin at different temperatures

T (°C)	Equations	R^2_{adj}
	Flow behavior index	
25	$n= - 0.52\ C_{XG} + 0.03\ C_P + 9.3x10^{-3}\ C_W$	0.979
50	$n= - 0.31\ C_{XG} + 0.08\ C_P + 7.9x10^{-3}\ C_W$	0.892
75	$n= - 0.36\ C_{XG} + 0.02\ C_P + 9.8x10^{-3}\ C_W$	0.661
	Consistency index (Pa sⁿ)	
25	$K= 381.54\ C_{XG} - 107.36\ C_P - 0.04\ C_W - 2.62\ C_{XG} C_P - 3.82\ C_{XG} C_W + 1.11\ C_P C_W$	0.986
50	$K= 106.92\ C_{XG} +121.37\ C_P + 0.04\ C_W - 1.78\ C_{GX} C_P - 1.07\ C_{XG} C_W - 1.26\ C_P C_W$	0.929
75	$K= 140.46\ C_{XG} -5.19\ C_P + 2.0x10^{-3}\ C_W - 0.58\ C_{XG}\ C_P - 1.42\ C_{XG} C_W + 0.05\ C_P C_W$	0.739

T - temperature (°C), R^2_{adj} - adjusted coefficient of determination; C_{XG} - xanthan gum concentration, C_P - pectin concentration and C_W - water concentration. All concentrations were expressed in % (w/w)

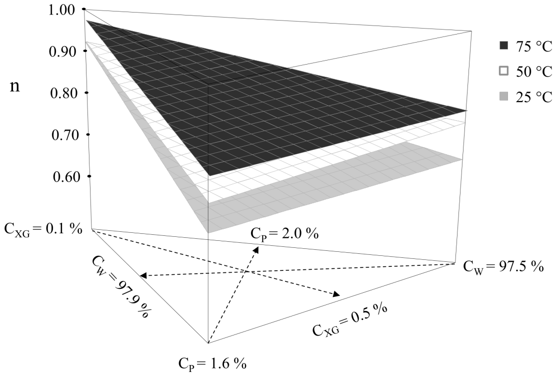


Fig. 3. Flow behavior index (n) of aqueous xanthan gum-pectin mixtures as a function of temperature and concentration of xanthan gum (C_{XG}), pectin (C_P) and water (C_W).

3.5 *Flow behavior index (n)*

The predicted flow behavior index for mixtures is presented in Fig. 3. The arrows shown in the lower part of the figure indicate how to move along the concentration axes to locate and read the n values. For example, at 75 °C, a solution containing 1 % xanthan gum, 2.0 % pectin, and 97.9 % has a behavior index of 0.98.

The n values of the mixtures varied from 0.70 to 0.92, 0.76 to 0.92 and 0.82 to 0.98 at 25, 50 and 75 °C, respectively. According to the flow behavior index values ($n < 1$), the aqueous xanthan gum-pectin mixtures had a predominating pseudoplastic character, which emphasizes the significant influence of the xanthan gum, despite being in lower concentration.

It has been observed that the thickening power of hydrocolloid compounds such as pectin and xanthan gum depends on its concentration and molecular weight (Saha and Bhattacharya, 2010). Therefore, as the xanthan gum has a molecular weight of around 6000 kDa (Argin-Soysal *et al*., 2006), it would have a more significant role on the rheological behavior of the mixtures than pectin, which has a molecular weight between 50 and 150 kDa (Sriamornsak, 2003). Similar effect of xanthan gum was observed in mixtures of xanthan gum-rice starch (Kim and Yoo, 2006).

Considering the n values of the xanthan gum solutions (0.24 - 0.63, Table 3), the pectin-xanthan gum mixtures exhibit a lower pseudoplastic character, since n ranged from 0.70 to 0.95. This could be related to a reduction in the interaction of xanthan gum with water due to the presence of pectin in the mixture, wich has greater charge density (0.3 mol/mol) than xanthan gum (0.25 mol/mol) (de Jong and van de Velde, 2007).

Regarding the effect of temperature, the mixtures of xanthan gum-pectin approached to a Newtonian behavior (Fig. 3) as temperature increased. It could be hypothesized that at low temperatures (~25 °C) xanthan gum has an helical form but it would changes to an heterogeneous structural conformation at higher temperatures (> 55 °C) (Khouryieh *et al*., 2006; Casas *et al*., 2000).

3.6 *Consistency index (K)*

The consistency index, considered as an indicator of the viscous nature of the system (Simuang *et al*., 2004), increased with the concentration of the hydrocolloids and decreased with rising temperature (Fig. 4). Both factors influence in opposite direction the intermolecular forces, whose extent is determined directly by the intermolecular spacing and the hydrogen-bonds strength (Azoubel *et al*., 2005). A similar effect was observed in mixtures of tapioca starch-pectin-carrageenan and flaxseed gum-native maize starch (Babić *et al*., 2006; Wang *et al*., 2008). It should be noted that higher xanthan gum concentrations imply lower pectin concentrations in the mixture.

The mixtures consistency index varied from 0.04 - 0.75 Pa s^n, over the studied temperature range. There is an important difference between these K values and those obtained for the xanthan gum solution at the concentration of 0.5 % (Table 3, Fig. 4). At this concentration, xanthan gum is essentially a pseudoplastic fluid, but this characteristic is attenuated when mixed with pectin. A higher consistency index reduction was observed in tomato ketchup after addition of pectin (Gujral *et al*., 2002). This change may be partially ascribed to the weakened potential of the gum to absorb water, as a result of the pH change.

The aqueous mixtures of xanthan gum and pectin could be used in edible films elaboration (Baümler *et al*., 2013), food and pharmaceutical formulation (Gujral *et al*., 2002; Sriamornsak, 2003) and as encapsulating material (Sansone *et al*., 2011; Rutz *et al*., 2013).

3.7 *Activation energy of flow (E_a)*

The temperature sensitivity of the consistency index of the mixtures was assessed by the Arrhenius model (Eq. 3). The resulting Ea values of the 16 blends were analyzed with the Design-Expert 7 software, and

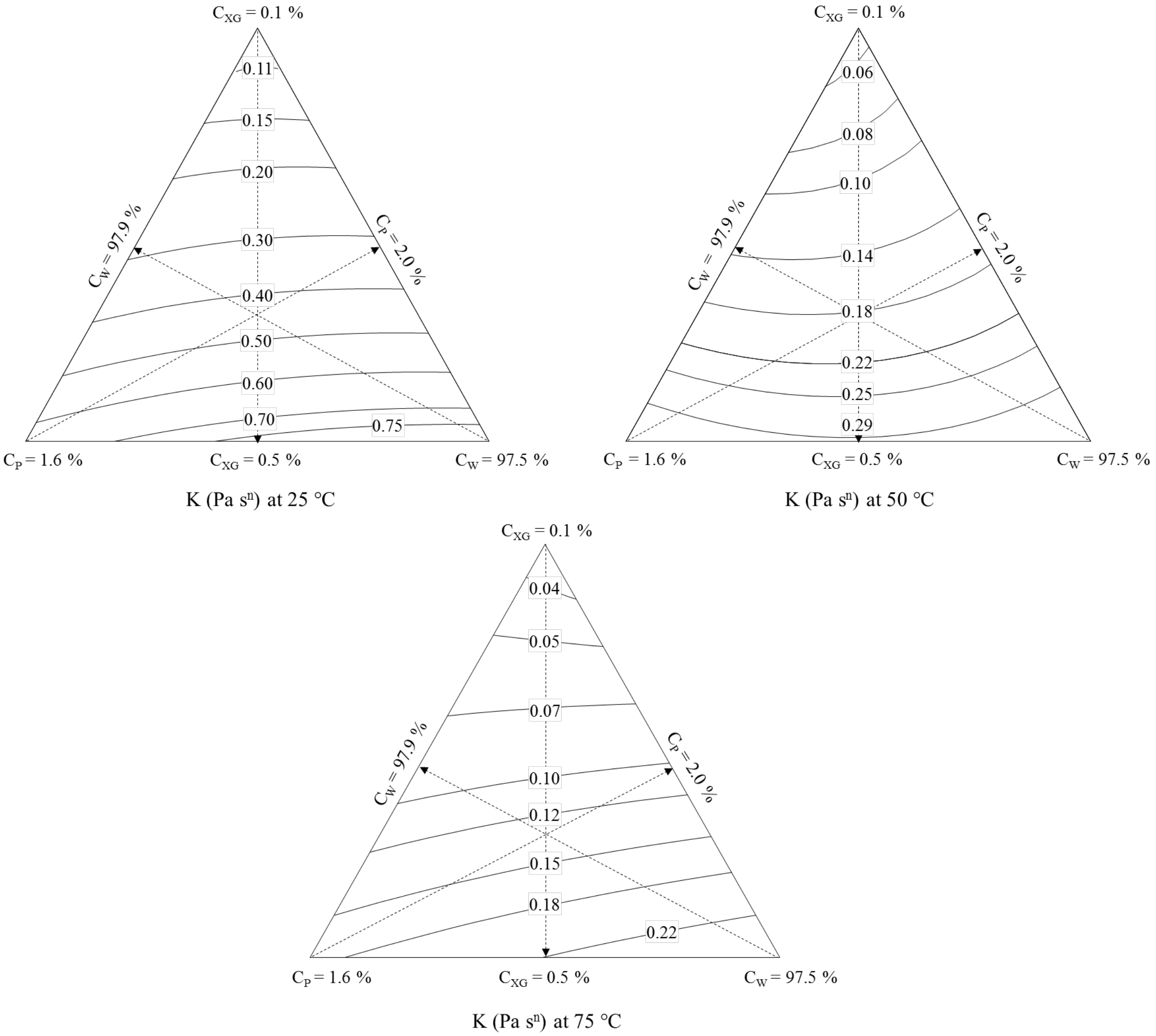


Fig. 4. Consistency index (K) of aqueous xanthan gum-pectin mixtures as a function of temperature and concentration of xanthan gum (C_{XG}), pectin (C_P) and water (C_W).

a polynomial model was developed for predicting E_a as a function of the blend composition (Eq. 4, $R^2_{adj}=$ 0.950). The linear terms and the interactions $C_{XG}-C_P$ and C_P-C_W showed a significant effect ($p < 0.05$) on E_a. The activation energy of flow was not significantly (p > 0.05) influenced by the $C_{XG}-C_W$ interaction.

$$\begin{aligned} E_a =& 1731.1C_{XG} - 14407.4C_P - 6.6C_W - 92.9C_{XG}C_P \\ & - 13.1C_{XG}C_W + 150.5C_PC_W \end{aligned} \tag{4}$$

where E_a is the activation energy of flow (kJ mol^{-1}) and C_{XG}, C_P, and C_W are the respective concentration of xanthan gum, pectin, and water (%).

This polynomial model is an important engineering tool since in combination with the Arrhenius relationship (Eq. 3) allows to determine the consistency index of the xanthan-pectin mixtures at a particular composition and temperature, within the studied experimental conditions.

The predicted activation energies of xanthan gum-pectin mixtures ranged from 17 to 25 kJ mol^{-1} (Fig. 5). These E_a values were higher than those reported for xanthan gum-rice starch mixtures (3.77 - 7.53 kJ mol^{-1}) (Kim and Yoo, 2006). A decrease in the Ea of the blends with increasing xanthan gum concentration

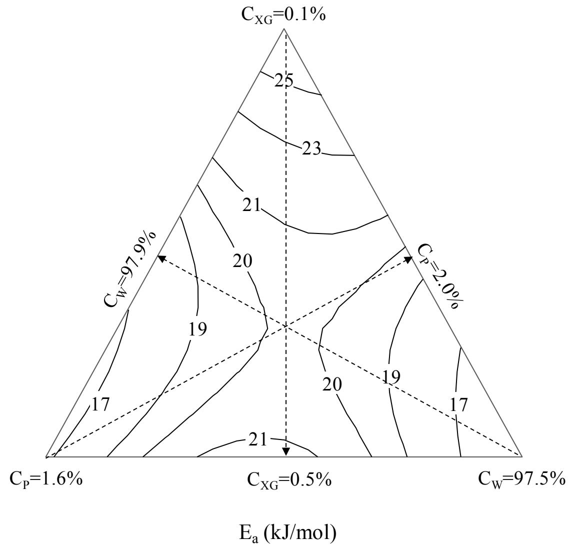


Fig. 5. Activation energy of flow (E_a) of aqueous mixtures of xanthan gum-pectin as a function of concentration of xanthan gum (C_{XG}), pectin (C_P) and water (C_W).

was observed, which is consistent with the reported thermal stability of the xanthan gum (Choi and Yoo, 2009). According to the high energy of activation, the xanthan gum-pectin mixtures are more temperature-dependent than the individual solutions of the gum (Table 3).

Conclusions

The aqueous xanthan gum-pectin mixtures showed a pseudoplastic behavior, described by the power law model. The behavior and consistency indexes of xanthan gum solution were influenced by concentration but not by temperature. Temperature dependency of the pectin viscosity solutions and consistency indexes of the solutions of xanthan gum/pectin-gum mixtures followed the Arrhenius model. Polynomial models were developed to predict the behavior index and the consistency index of the xanthan gum-pectin solutions as a function of composition, at a given temperature. Quantitative relationships between the activation energy and concentration and temperature of the mixtures were developed.

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