Viscoelastic and Textural Characteristics of Masa and Tortilla from Extruded Corn Flours with Xanthan Gum

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1. Introduction

Corn plays an important role in the diet of many countries, especially México (Serna-Saldivar et al 1990), where corn tortilla is the principal ancestral food, and its consumption per capita is approximately 120 kg (Martínez et al. 2004). Corn tortilla can be produced with fresh masa using the traditional nixtamalization process, or with instant nixtamalized corn flour (Arámbula et al. 1999). Both processes result in industrial effluents known as nejayote (Serna-Saldivar 1996). An alternative process to avoid such contamination is extrusion which is defined as a continous process in which mechanical cutting is combined with heat to obtain gelatinization of the starch and denaturation of the proteins. It yields a plastified and restructured product with new shapes and textures. Some of the characteristics for which extrusion has gained popularity are versatility, reduced costs, high productivity, high-quality products, different product shapes, energy efficiency, and generation of new products and absence of effluents (Harper 1989).

Extrusion has been utilized as a continuous process of nixtamalization to produce instant flour, then to make corn tortillas, giving good product such as that of fresh masa obtained from the traditional process (Arámbula et al. 1998; Arámbula et al. 2002; Galicia 2005; González 2006; Martínez-Flores et al. 1998; Milán-Carrillo et al. 2006; Reyes-Moreno et al. 2003). However, during its storage, tortilla became hard and diminishes in flexibility. This can be due to fact that after its preparation starch reorganizes, and as a consequence produce crystalline structures, which is known as retrogradation, altering the texture and nutritional characteristics of tortillas (Aguirre-Cruz et al. 2005).

To improve and preserve the quality of tortillas made from nixtamalized corn flour during their storage, new alternative have been studied. Among these alternatives is the use of some additives such as hydrocolloids which are water-soluble, high-molecular weight heteropolysaccharides. These compounds vary in shape and function and add flexibility to the tortillas produced, acting as a fat replacement, water binder, texturizer and adhesive. Among the main hydrocolloids utilized in tortillas is carboxymethylcelullose (CMC), guar gum, alginates, carrageenans and xanthan gum (Gurkin 2002).

Roman-Brito et al (2007) studied the effect of xanthan gum on nixtamalized corn flour with 0.25%(w/w), 0.50%(w/w), and 0.75%(w/w) of xanthan gum to make tortillas. These authors observed a decrease in hardness and increase in flexibility in the tortillas during their storage at 4°C. Yau et al. (1994) also extended the stability of tortillas made from corn flour during their storage at 25°C with xanthan gum at 1% (w/w), along with other additives. Arämbula et al. (1999), prepared tortillas with extruded corn flour with the addition of hydrocolloids: CMC, arabic gum, guar gum and xanthan gum at 0.5% (w/w) with different concentrations of lime. Tortillas were obtained with good results regarding to their textural characteristics (rollability, extensibility and shear force) with masa containing 0.2 %(w/w) of lime and xanthan gum added before extrusion. The unique structure and properties of xanthan gum account for its potential in versatile applications in the food industry (Hanna et al. 1997). The effect of hydrocolloids has not been studied at any concentration or mixture of them in the production of tortillas from extruded corn flour. It could help to keep a soft texture of tortillas during storage. The addition of hydrocolloids can modify the rheological (viscoelasticity) properties of corn masa, which are important in the tortilla making process. The understanding of these parameters is crucial for the design of equipment and definition of operation parameters in the tortilla industry (Aguirre-Cruz et al. 2005).

On the other hand, one of the methods most frequently utilized for the study of viscoelastic properties of masa is the dynamic method. In this test, results obtained refer to parameters that help to characterize appropriately the materials whose rheological behavior is complex, such as dynamic moduli of storage and loss, respectively the energy stored elastically and that dissipated as heat during a cycle of deformation (Faubion and Hoseney 1990).

The storage modulus G' is an indicator of the elastic component of the material, and the loss modulus G'' is an indicator of the viscous component. The storage and loss moduli are usually reported as a function of frequency. The phase angle represents a simple mean of elastic and viscous natures of the material. On some occasions, this property is expressed as the tangent of the phase angle (Tan δ), that is, the ratio between the loss and storage moduli (G''/G'). Moisture content is an important element in the determination of viscoelastic properties of cereals, which are reduced proportionally with moisture content (Masi et al. Measurement of viscoelastic characteristics have been utilized in dispersions in masas of nixtamalized corn dehydrated with hydrocolloids (Aguirre-Cruz et al. 2005), and of commercial corn flour at different moisture content: 35%, 40% and 50%, respectively (Broulliet-Fourmann et al. 2003).

The aim of this research was to determine the effect of xanthan gum on viscoelastic and textural characteristics of masa and tortilla from extruded nixtamalized corn flour, and to find the best combination of extrusion process factors to produce corn flour with xanthan gum to make tortillas with the best texture. We use response surface methodology (RSM).

2. Experimental procedure

2.1. Raw material

White corn (Dekalb variety) from Sinaloa de Leyva, Sinaloa, México was used. Corn was cleaned using a vibrating cleaner (Blount, model M2BC. Bluffon, IN, USA), and stored at 5 °C until used.

2.2. Extrusion process

Samples of corn (2 kg) were ground in a mill (Pulvex, model 200, Mexico, D.F.), with a 0.8-mm sieve. The ground corn was mixed with commercial grade lime ($Ca (OH_2)$) at 0.3 % (w/w) and xanthan gum (Spectrum Chemical, Gardena, CA, USA) at different concentrations (0.3-0.7 % of corn weigth) and using an industrial mixer (Hobart, model AS200T. Troy, Ohio, USA). Next, distilled water was added to this blend up to reach the moisture content appropriated (range 25-35 % of the corn weight). To obain a complete hydration of the ground corn particles, samples were packed in a polyethylene bag and stored for 12 h at 5 °C. Before extrusion, samples were tempered at 25°C during 4 h.

The extrusion process was carried out in a single-screw laboratory extruder (Brabender, model 837416. Duisburg, Germany) with a 19 mm screw-diameter, length-to-diameter 25:1, nominal compression ratio 1:1, and a die opening of 3 mm. The first three zones of the extruder were maintained at 60 ºC, and the fourth zone was varied (110-130 °C), with a screw speed of 112 rpm. A screw-operated feed hopper fed the extruders at 45 rpm. Extrudates were dried at 60 °C for 1 h in a tunnel type dryer (no brand), and cooled at room temperature (25°C). To obtain the extruded nixtamalized corn flour (ENCF), the extrudates were ground in a mill (Pulvex, model 200, Mexico, D.F.) with a sieve 0.8-mm diameter, and packed in plastic bag at 5°C.

2.3. Corn flours evaluation

The ENCFs were analyzed for water absorption index (WAI) and subjective water absorption capacity (WAC). These response variables are the most critical for making tortillas at commercial level. WAI was measured using the method of Anderson et al. (1969) with a modification: The distilled water temperature was 25 °C instead of 30 °C, and theWAI was expressed as g of gel / g of dry matter. WAC was determined using the method described by Flores-Farías et al. (2002). The quantity of water added was recorded as the capacity for water absortion of the flour in mL of water / 100 g of flour.

2.4. Corn masa viscoelasticity

Corn masa was prepared from each ENCF using 100 g sample and adding distilled water. The quantity of water utilized corresponded to the WAC. Once prepared masas, they were allowed to stand for 30 min in a plastic bag at room temperature (25 °C). Samples of 2 g of masa each were weighed out to be utilized in the rheometer.

The oscillatory dynamic scanning test was performed utilizing a dynamic mechanical spectrometer (Rheometrics Scientific, model RSF III. Piscataway, NJ, USA) equipped with parallel plates of 25 mm diameter and a chamber for temperature control (peltier). A sample was placed between the plates separated by a gap of 2.5 mm. The excess of masa was cut off using a plastic instrument. Next, petroleum jelly was applied where the sample was air exposed to prevent loss of moisture. The frequency sweep test was carried out using a software (RSI Orchestrator, Rheometrics Scientific). Each test was run to a deformation of 0.04% and at 25°C, which gave a minimum of structure disorder and with sufficient assurance of the level of torsion (Broulliet-Fourmann et al. 2003). The deformation used was previously determined to work in the viscoelastic linear region in a frequency range from 0.1 to 100 rad/s. The viscoelastic parameters obtained in the frequency range used were the storage modulus (G') and loss modulus (G'') in kPa, and the tangent of the phase angle (Tan δ).

2.5. Corn tortilla preparation

Two kg of masa from each ENCF were mixed with distilled water to obtain masa. The amount of water was based on the WAC of each extruded treatment. Masas from ENCF were transported to a commercial factory. A roller machine (Rodotec, model RT-100, Guadalupe, N.L., México) was used with a mold of 14 cm diameter, and was adjusted to a masa weight of 25 g. Tortillas were baked in an oven (integrated to the roller machine) with 3 temperature zones: zone 1, 270 ± 10 °C; zone 2, 320 ± 30 °C; and zone 3, 300 ± 25°C, with a residence time of 60 s. The baked tortillas were cooled at room temperature (25 °C).

2.6. Corn tortilla textural evaluation

To determine firmmess and rollability, tortillas packaged in plastic bags were placed at room temperature (25°C). Firmness and rollability were measured at 2 h, 24 h and 48 h after baking. The firmness test was carried out according to the procedure reported by Ramírez-Wong et al. (2007), modifying the cross head speed of the texturometer (Instron, model 4465. Canton, MA, USA) to 50 cm/min. Firmness was expressed as maximal force (MF) in kPa. Regarding tortilla rollability, three strips 2 cm wide were cut from each tortilla and individually tested (Waniska 1976). Each strip of tortilla was rolled up in a wooden cylinder 2 cm in diameter, and was examined for degree of rupture, which was established on a scale of 1 to 5, where 5 indicated no tear of the tortilla (maximum rollability), 3 partial tear, and 1 complete tearing. Five tortillas were used for each treatment in each test.

2.7. Experimental design and statistical analysis

Response surface methodology (RSM) was used, and the process variables were: temperature of the fourth zone of the extruder (T, 110-130 °C), moisture content of conditioned ground corn (MC, 25-35 % of the corn weight), and concentration of xanthan gum (XG, 0.3-0.7 % of corn weigth). A central composite rotatable design was utilized (Table 1), with three factors and 5 levels (Montgomery 2001; Myers 1971). In order to observe the difference between specific treatments, Tukey's test was used at a level of significance of p= 0.05 . To find the best variables combination of the process to obtain the extruded corn flour, the conventional graphical method was used and maximizing WAI, Tan δ; minimizing G', G'' and MF. To obtain contour plots for the visualization and selection of the best combination of T, MC and XG to prepare the extruded nixtamalized corn flour, the contour plots of each of the response variables were utilized, by means of the method of superimposition of surfaces. Data analysis and the elaboration of surface response and contour plots were performed using Design Expert version 6.0.7 software (Design Expert, 2002).

3. Results and discussion

3.1. Corn flours evaluation

WAI is a parameter that gives an idea of the absorption of water of corn flour, and is an indicator of yield of fresh masa (Molina et al. 1977). The highest value of WAI in ENCFs was of 3.6 g of gel/g of dry matter, and was obtained at high concentration (0.84%) of xanthan gum (treatment 14, Table 1), which would indicate the capacity of the gum to form gels. This could be due to the high affinity of hydrocolloids for water, because of its branched structure. During hydration, water molecules hydrogen bond with hydroxyl (or carboxyl) groups found in the unit components (sugars) of hydrocolloid molecules, inducing this association with increased capacity for water retention (Dickinson 2003). Aguirre-Crus et al. (2005) observed in their research an increase in the capacity for water retention in suspensions of masa of corn dehydrated with hydrocolloids at different temperatures.

WAC is the quantity of water that is absorbed by the flour to obtain a masa of appropriate consistency for the preparation of tortillas and is a subjective test. WAC was affected very significantly ($p < 0.01$) By the treatment. The WAC range in the ENCFs was between 74.8 - 89 mL water/100 g flour (Table 1). Arámbula et al. (1999) reported in extruded flours with xanthan gum a high WAC value of 88.5 mL/100g. However, the value for extruded flour reported by González (2006) was 72 mL water/100 g flour, which was low; probably due to that none type of gum was added. Arámbula et al. (2002) found in extruded flour a WAC of 70 mL/100g with 0%, and 80 mL/100g with 3% of addition of corn pericarp, respectively.

Treatment[a]	Process Factors[b]			Response Variables[c,d]					
	T(X_1)	MC (X_2)	XG(X_3)	WAC	WAI	G'	G''	Tan δ	MF
1	110 (-1)	25 (-1)	0.30 (-1)	76.9	3.0	274.3	58.3	0.21	53.3
2	130 (+1)	25 (-1)	0.30 (-1)	77.0	2.8	269.3	59.8	0.22	59.0
3	110 (-1)	35 (+1)	0.30 (-1)	76.3	3.2	223.0	54.8	0.25	47.0
4	130 (+1)	35 (+1)	0.30 (-1)	78.6	3.3	302.0	60.0	0.20	48.5
5	110 (+1)	25 (-1)	0.70 (+1)	74.8	2.6	231.0	51.6	0.22	58.5
6	130 (-1)	25 (-1)	0.70 (+1)	79.6	2.2	270.3	63.3	0.23	58.6
7	110 (-1)	35 (+1)	0.70 (+1)	79.0	3.4	213.3	48.2	0.23	54.3
8	130 (+1)	35 (+1)	0.70 (+1)	79.6	3.2	234.0	51.2	0.22	41.8
9	103.18 (-1.681)	30 (0)	0.50 (0)	82.8	3.5	231.0	50.7	0.22	44.9
10	136.82 (+1.681)	30 (0)	0.50 (0)	76.6	3.4	219.3	52.7	0.24	51.0
11	120 (0)	21.59 (-1.681)	0.50 (0)	80.1	2.5	257.0	61.6	0.24	61.3
12	120 (0)	38.41 (+1.681)	0.50 (0)	79.0	2.9	294.7	72.1	0.24	55.9
13	120 (0)	30 (0)	0.16 (-1.681)	83.1	3.0	243.7	50.7	0.21	49.7
14	120 (0)	30 (0)	0.84 (+1.681)	89.0	3.6	211.0	47.8	0.23	43.7
15	120 (0)	30 (0)	0.50 (0)	87.3	3.4	218.7	54.7	0.25	46.0
16	120 (0)	30 (0)	0.50 (0)	87.0	3.2	190.3	45.7	0.24	43.6
17	120 (0)	30 (0)	0.50 (0)	87.3	3.5	225.3	55.8	0.25	46.5
18	120 (0)	30 (0)	0.50 (0)	87.0	3.5	218.7	49.3	0.23	49.1
19	120 (0)	30 (0)	0.50 (0)	87.3	3.2	176.6	46.5	0.26	41.2
20	120 (0)	30 (0)	0.50 (0)	87.0	3.1	210.3	53.5	0.25	41.2

[a]Numbers do not correspond to the order of processing. [b]T, extrusion temperature (°C); MC, moisture content (%, w/w); XG, xanthan gum (%, w/w); values in parentheses are the coded levels. [c]WAC, subjetive water absorption capacity (mL water/100/g flour; WAI, water absorption index (g gel/g dry matter); G', storage modulus (kPa), G'', loss modulus (Kpa); Tan δ, tangent of the phase angle; MF, maximal force (kPa). [d]Mean of three replicates.

Table 1. Experimental designa used to obtein different combinations of extrusion temperature/moisture content/xanthan gum for production of extrusion-nixtamalized corn flour.

3.2. Masa viscoelasticity evaluation

In Figures 1(a, b), 2(a, b) and 3(a, b) are presented the storage modulus (G'), loss modulus (G'') and tangent of the phase angle (Tan δ), respectively. It is observed that all of these viscoelastic parameters increased with frequency. The range 0.1-10 rad/s was the most susceptible to structural changes (Broulliet-Fourmann et al. 2003). In this frequency range, there was a considerable increase in the viscoelastic parameters G', G'' and Tan δ, whereas small gradual changes occurred at high frequencies. Hence, the values of the viscoelastic parameters of 10 rad/s were selected for the optimization of the variables of the process for making tortillas from ENCFs. Values of G' were higher than those of G'' (Table 1), indicating that the elastic behavior predominated over the viscous behavior of masas. Similar trend was found by Aguirre-Cruz et al. (2005) in samples of suspension of corn masa at 10% (w/w) of solids in their heating/cooling kinetics, and by Broulliet-Fourmann et al. (2003) in corn flours at different moisture contents (35%, 40% and 50%).

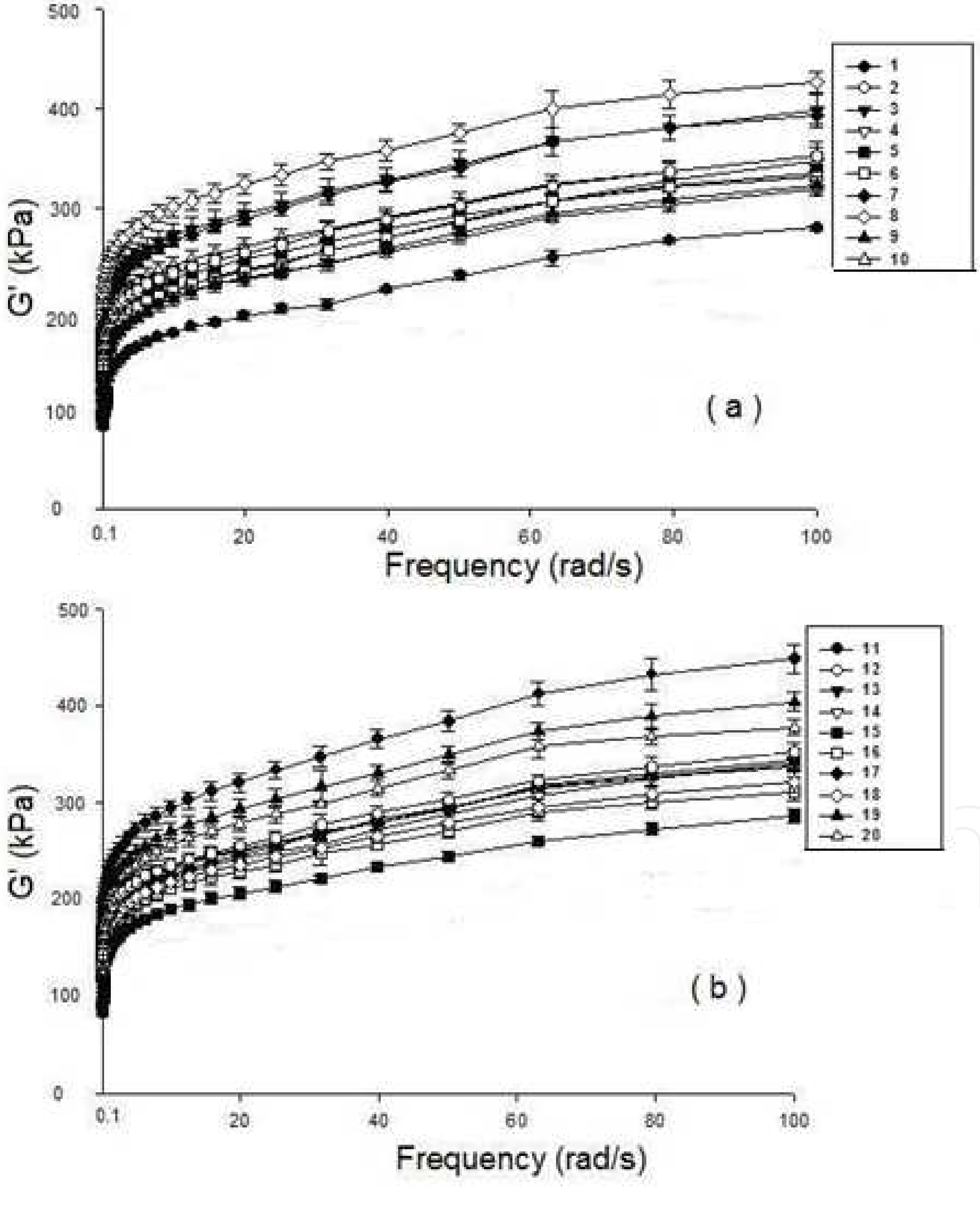


Figure 1. Storage modulus (G') vs frequency for extruded nixtamalized corn flours in treatments: (a) 1-10 and (b) 11-20. Error bars indicate standard error of means.

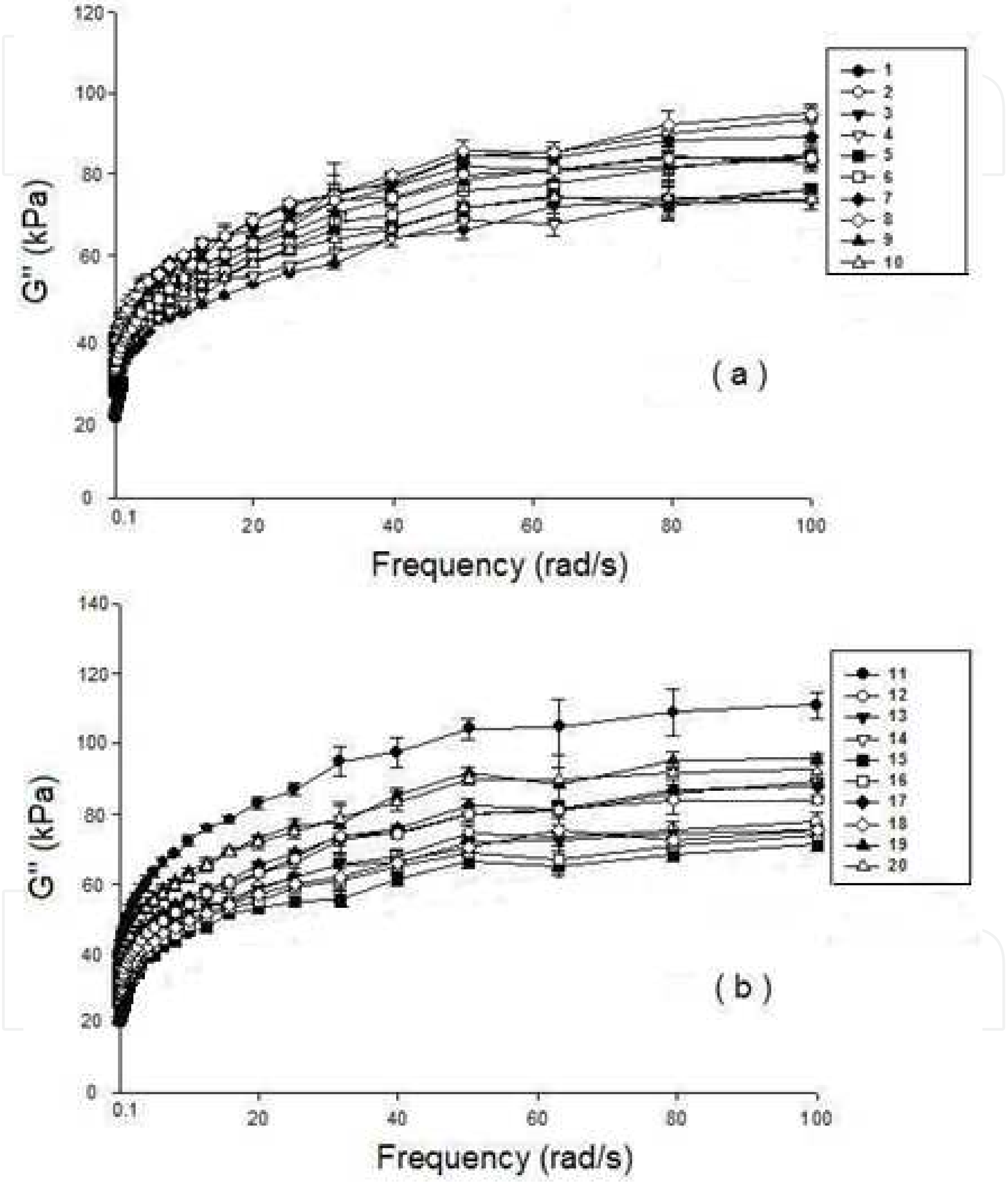


Figure 2. Loss modulus (G'') vs frequency for extruded nixtamalized corn flours in treatments: (a) 1-10 and (b) 11-20. Error bars indicate standard error of means.

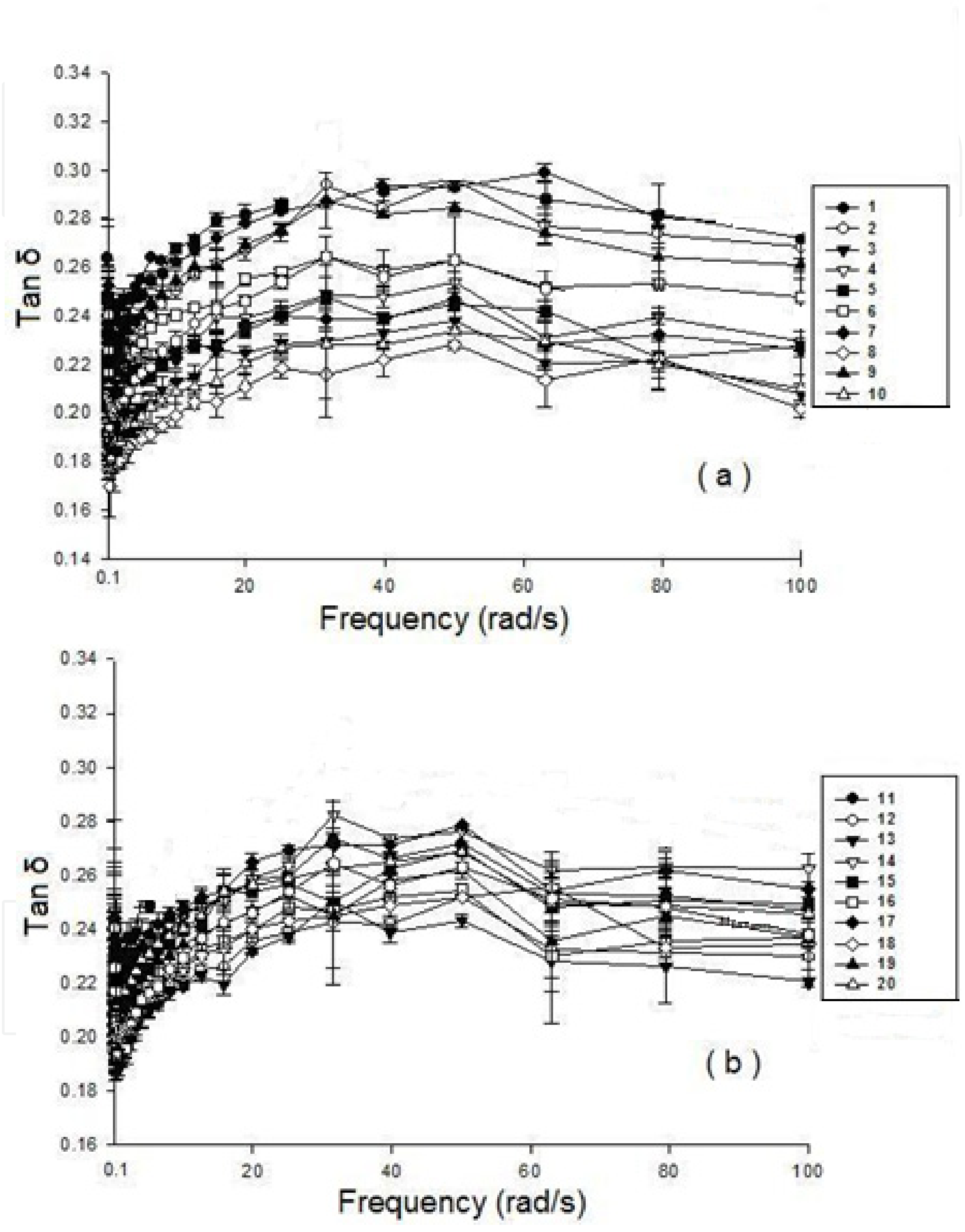


Figure 3. Tangent of the phase angle (Tan δ) vs frequency for extruded nixtamalized corn flours in treatments: (a) 1-10 and (b) 11-20. Error bars indicate standard error of means.

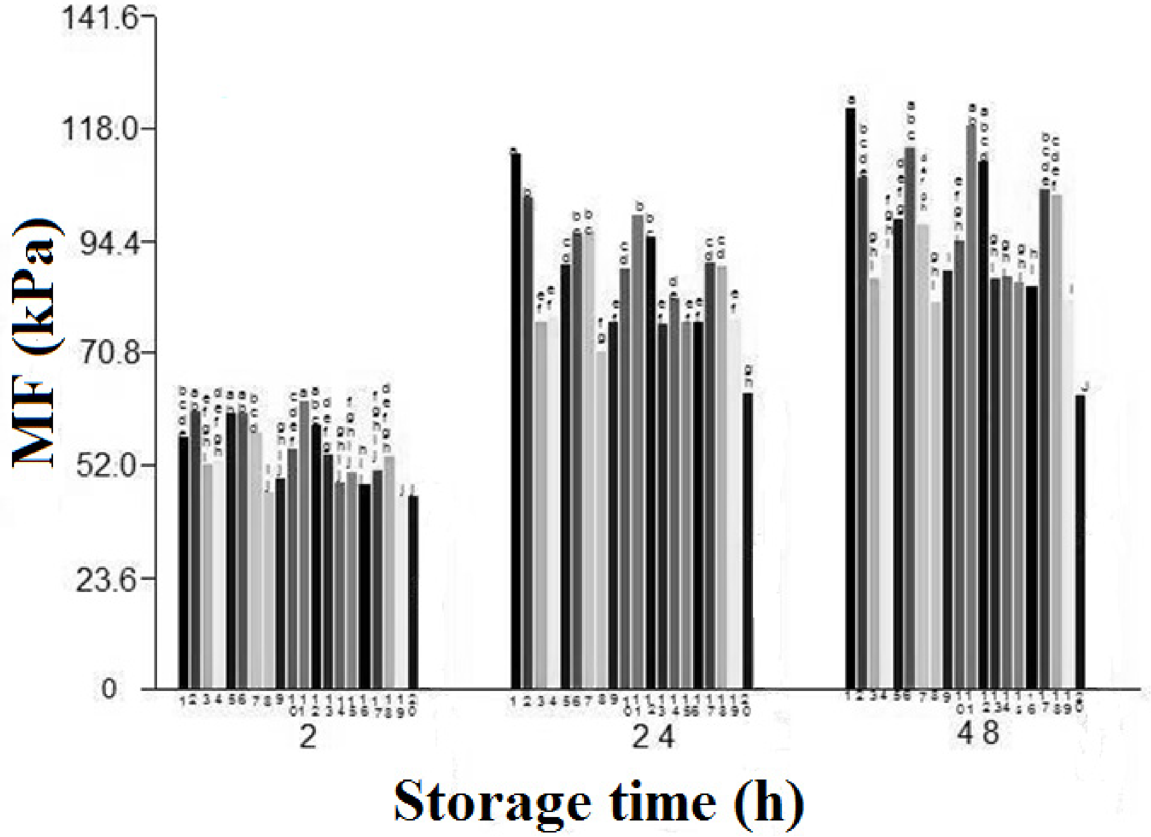
In the storage modulus (G'), the lowest value (176.6 kPa) was with treatment 19 (Table 1) with no significant difference ($p < 0.05$) with treatment 16, whose conditions were the same. When the concentration of xanthan gum increased, at the same conditions of temperature and moisture content, G' decreased. It probably was due to that the water molecules and bound components (amylose-hydrocolloid) of masas with hydrocolloids formed a gel with a softer structure, which affected G' values (Aguirre-Cruz et al. 2005). Regarding to the loss modulus (G''), the lowest value (45.7 kPa) was obtained for treatment 16 (Table 1), however, there were not significant diferences ($p < 0.05$) with treatments 7, 14 and 19. The highest value of Tan δ was observed in treatment 19 with 0.26 (Table 1), which differed significantly ($p < 0.05$) to the rest of the treatments. Since Tan δ values for all masas were in the range of 0.2-0.3, it is corresponding to that of an amorphous polymer (Ferry 1980). Similar values were obtained by Aguirre-Cruz et al. (2005) during cooling of diluted corn masa.

3.3. Corn tortilla evaluations

Values for the physical characteristics of tortillas from ENCFs after two hours they were made, time at which they are eaten, were similar to those of commercial tortillas. The physical characteristics evaluated were: weight, (range 21.3-25.2 g); diameter (range 12.2-12-7 cm); and thickness (range of 1.4-1.9 cm). Similar results were reported by Ramirez-Wong (1989) who evaluated corn tortillas obtained by the traditional process of nixtamalization.

Figure 4 presents the tortilla firmness as maximum force (MF) to rupture the tortillas made from the different ENCFs after 2 h, 24 h and 48 h of storage at room temperature (25°C). In general, for all the treatments (Table 1), as time progressed tortilla firmness increased. At 2 h of storage time, the lowest MF (41.2 kPa), which is the best value for tortilla firmness (softer) were observed in treatments 8, 9, 14, 15, 16, 17, 19 and 20 (Table 1), and they were not significantly different ($p > 0.05$). At 24 h of storage time the lowest MF (62.8 kPa) was observed for treatment 20. At 48 h of the storage time the lowest value (62.3 kPa) was similar to that of 24 h, corresponding to the same treatment (20), where there was a significant difference ($p<.05$) compared to all other treatments. The lowest values of firmness in tortillas during storage corresponded to high concentrations of xanthan gum (0.5% to 0.84%). Treatments with high xanthan gum concentrations retained the tortilla moisture and improving its textural characteristics. This finding is similar to that reported by Arámbula et al. (1999), who obtained better results when utilizing xanthan gum compared to other addtitives such as guar gum, CMC and Arabic gum.

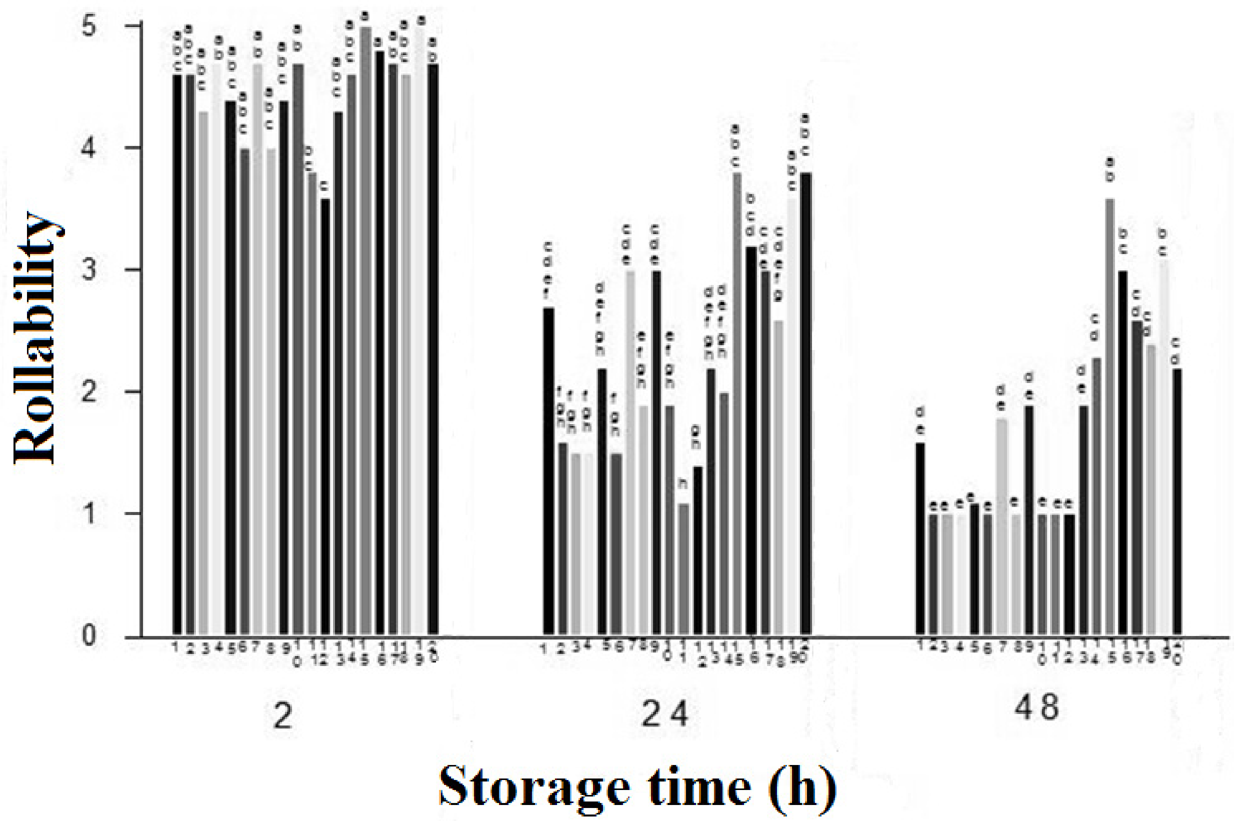
Tortillas made with extruded nixtamalized corn flours showed an increase in their MF during their storage at 25°C. Regarding to texture, the most important changes during storage occurred the first 24 h (Fernández et al. 1999; Ramírez-Wong 1989). Since some starch crystals are retained after baking of the tortilla, they serve as nuclei which facilitate the rapid association of starch molecules, and structural changes occur during the initial 24 h following baking, which in turn leads to rapid retrogradation or increasing the texture of this product (Fernández et al. 1999). A similar tendency for MF was obtained by Roman-Brito et al. (2007) for tortillas made with nixtamalized flour containing xanthan gum and stored for 3 days at 4°C; however, the increase was less due to the lower storage temperature. Results obtained for tortillas made from ENCFs are similar to those reported by Galicia (2005) and Gonzalez (2006) after 2 h of storage. Neverthaless, at 24 h and 48 h of storage the MF values obtained in the present study were lower than those reported for these authors because they did not use gum in masas preparation.



In each storage time, MF values with the same letter are not statistically significant (p > 0.05).

Figure 4. Effect of storage time on the maximum force (MF) to rupture for tortillas made from extruded nixtamalized corn flours.

Figure 5 presents tortilla rollability for all ENCF treatments after 2 h, 24 h and 48h of storage at room temperature (25°C). In general, for all the treatments (Table 1), as time progressed tortilla rollability decreased. For all ENCF treatments, the best tortilla rollability was obtained at the 2 h of storage. The most rapid loss of tortilla rollability occureed within 24 h of storage time, such as in the study of tortillas made from corn flour with xanthan gum added and stored at 4°C by Roman-Brito et al. (2007). It could be due to the very rapid association (retrogradation) of amylose and of amylose and amylopectin in starch (Fernández et al. 1999). At 2 h of storage time, the highest rollability (a value of 5) was obtained for all treatments, except for 11 and 12 (Table 1). At 24 h of storage, treatments 15, 19 and 20 showed the highest rollability values (3.6-3.8). For 48 h of storage, treatment 15 showed the highest value (3.6). The highest values for rollability during storage corresponded to the xanthan gum concentration of 0.5%, which offers greater flexibility to the tortillas as in the study made by Arámbula et al. (1999), who used the same concentration and the same gum.



In each storage time, rollability values with the same letter are not statistically significant ($p > 0.05$).

Figure 5. Effect of storage time on the rollability of tortillas made from extruded nixtamalized corn flours.

3.4. Extrusion process optimization

To find the best variables combination of the process (T, MC and XG) to obtain the extruded nixtamalized corn flour, response surface methodology (RSM) was used. The evaluations to optimize the extrusion process were: WAI, G′, G′′, Tan δ and MF.

Water absorption index. Analysis of variance showed that WAI of extruded flours depended on the linear term of the conditioning moisture content (MC, $p < 0.01$), quadratic term of MC [$(MC)^2$, $p < 0.01$] and combined term of moisture content with xanthan gum [(MC)(XG), $p < 0.10$]. The prediction model in terms of original variables for WAI was:

$$Y_{WAI} = -4.37 + 0.583MC + 0.137\left(MC\right)\left(GX\right) - 0.01\left(MC\right)^2 \quad (1)$$

The regression model explained 75% of the total variation ($p<0.01$) in WAI of extruded nixtamalized corn flours. Figs. 6 (a,b,c) show the effect of WAI extruded flours as a function of T, MC and XG, noting that an increase conditioning moisture content of flour increase the water absorption index, as it does the increase in xanthan gum concentration at high levels of moisture content. Vargas-López (1987) mentioned that extruded grits of corn-sorghum and corn starch exhibit a maximum WAI at high temperatures and high moisture content.

The highest value of WAI (3.34 g gel/g dry matter) was observed at T=110-120.94°C/MC=29.68-35% (w/w)/XG=0.48-0.70% (w/w) (Figs. 6 (a,b,c)). Similar values of WAI for extruded flours were obtained by Reyes-Moreno et al. (2003)

Storage modulus. The linear term of xanthan gum (XG, $p<0.01$) and quadratic term of moisture content [$(MC)^2$, $p < 0.01$) affected significantly G′ in masa of ENCFs. The prediction model in terms of original variables for G′ was:

$$Y_{G'} = 1035 - 64XG + 0.97(MC)^2 \tag{2}$$

The regression model explained 70% of the total variation ($p < 0.05$) in G′ of extruded nixtamalized corn flours. Figs. 7 (a,b,c) show the effect of G′ in masa of ENCFs as a function of T, MC and XG, observing that with any value of moisture content from approximately the central point (30%), G′ increaces, due to its quadratic effect, and that increasing the concentration of XG decreases G', because of its linear effect. The lowest value (214.58 kPa) was observed at T=110-130°C/MC=28-32.1% (w/w)/GX=0.57-0.70% (w/w) (Figs. 7 (a,b,c).

Loss modulus. The viscous modulus G'' in masa of ENCFs depended on the quadratic term of moisture content [$(MC)^2$, $p < 0.01$)]**.** The prediction model in terms of original variables for G′′ was:

$$Y_{G''} = 235 + 0.22(MC)^2 \tag{3}$$

The regression model explained 72% of the total variation ($p < 0.01$) in G′′ of masa from extruded nixtamalized corn flours. Figs. 8 (a,b,c) show the effect of G′′ in masa from ENCFs as a function ot T, MC and XG, noting that for any value of moisture content starting at the central point (30%), G'' increased due to the quadratic effect. The lowest value (49.35 kPa) was observed at T=110-128.32°C/MC=27.4-32.72% (w/w)/XG=0.52-0.70% (w/w)/ (Figs. 8 (a,b,c)).

Tangent of the phase angle. Analysis of variance showed that Tan δ of masa from ENCFs depended on the quadratic terms of temperature (T^2, $p < 0.05$) and xanthan gum [$(XG)^2$, $p < 0.01$], and on the interaction temperature-moisture content [$(T)(MC)$, $p < 0.05$)]. The prediction model in terms of orginal variables for Tan δ was:

$$Y_{\mathrm{Tan}\delta} = -1.59 - 0.0002(T)(MC) - 0.00007(T)^2 - 0.31(XG)^2 \tag{4}$$

The regression model explained 71% of the total variation ($p < 0.05$) in Tan δ of masa from extruded nixtamalized corn flours. Figs. 9 (a,b,c) show the effect of Tan δ of masa from ENCFs as a function of T, MC and XG, observing that at any concentration of xanthan gum from approximately the central point (0.5%), Tan δ decreases due to its quadratic effect. The highest value (0.239) was shown at T=116.61-124.55°C/MC=0.25-0.35% (w/w)/XG=0.47-0.59% (w/w) (Figs. 9(a,b,c)).

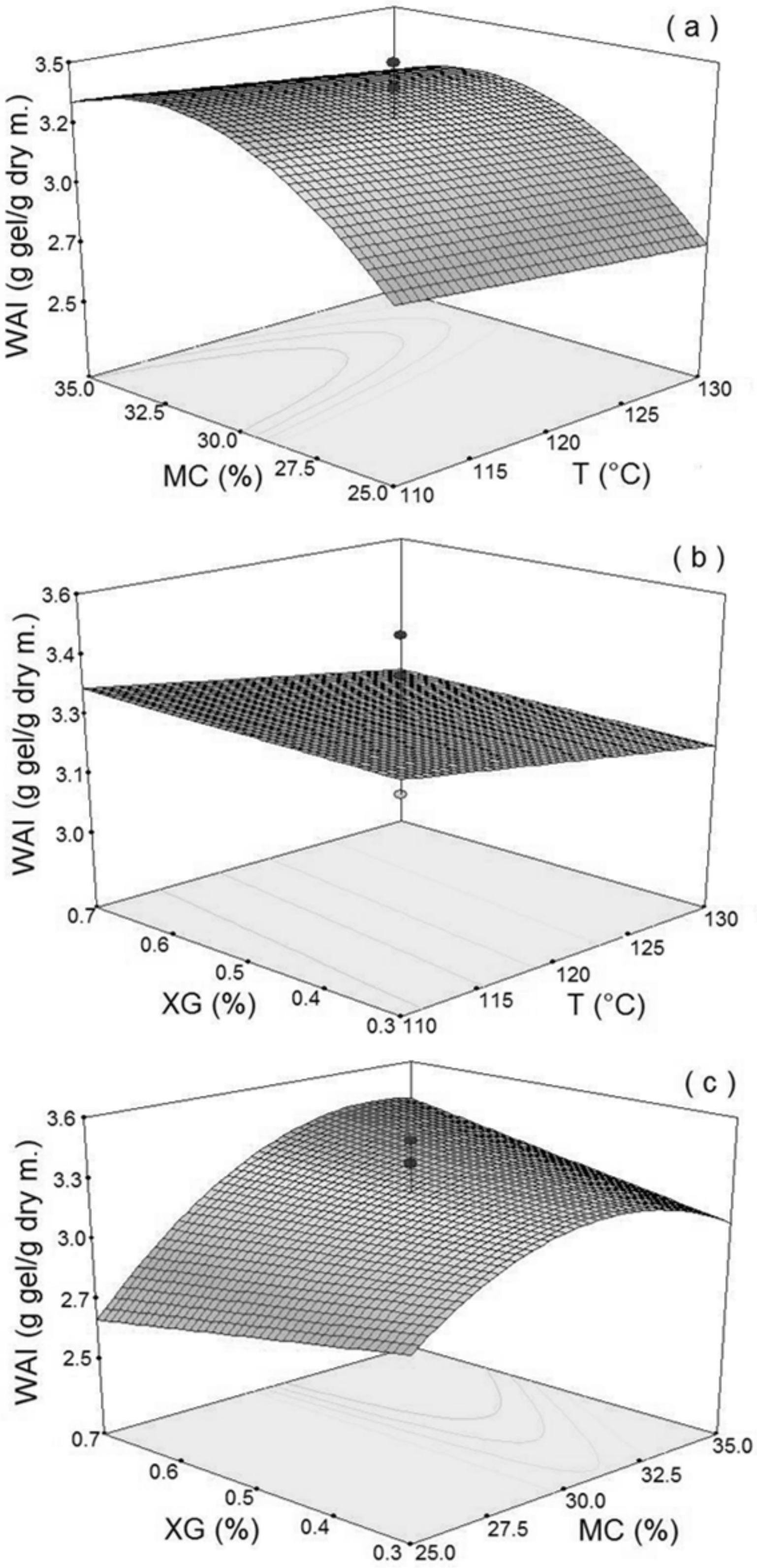


Figure 6. Response surface and contour plots for the effect of extrusion process factors on water absorption index (WAI) from extruded nixtamalized corn flour. (a) Effect of moisture content (MC, %) and extrusion temperature (T, °C) on WAI; (b) Effect of xanthan gum (XG, %) and T on WAI; (c) Effect of XG and MC on WAI.

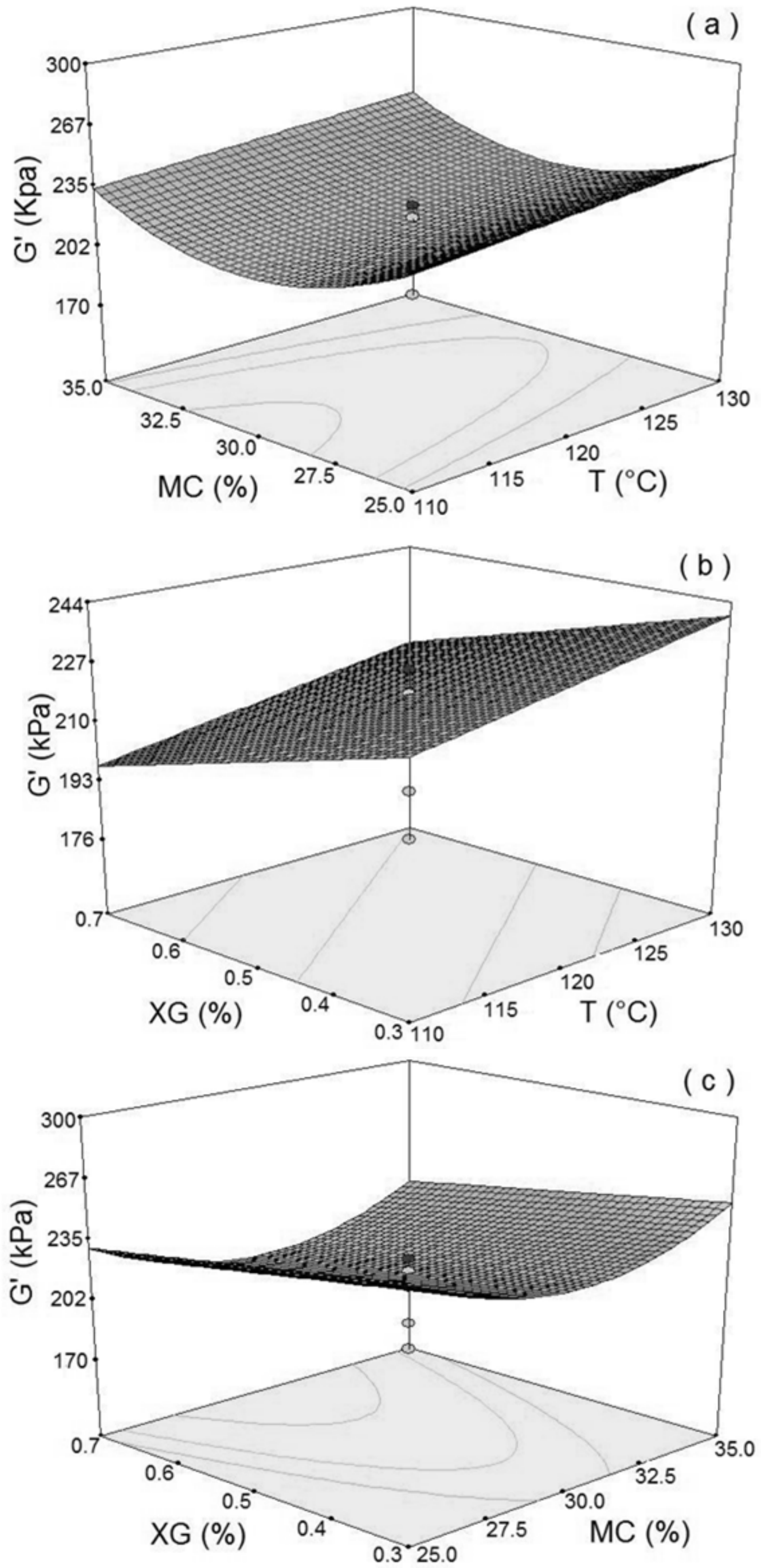


Figure 7. Response surface and contour plots for the effect of extrusion process factors on storage modulus (G') of masa from extruded nixtamalized corn flours. (a) Effect of moisture content (MC, %) and extrusion temperature (T, °C) on G'; (b) Effect of xanthan gum (XG, %) and T on G'; (c) Effect of XG and MC on G'.

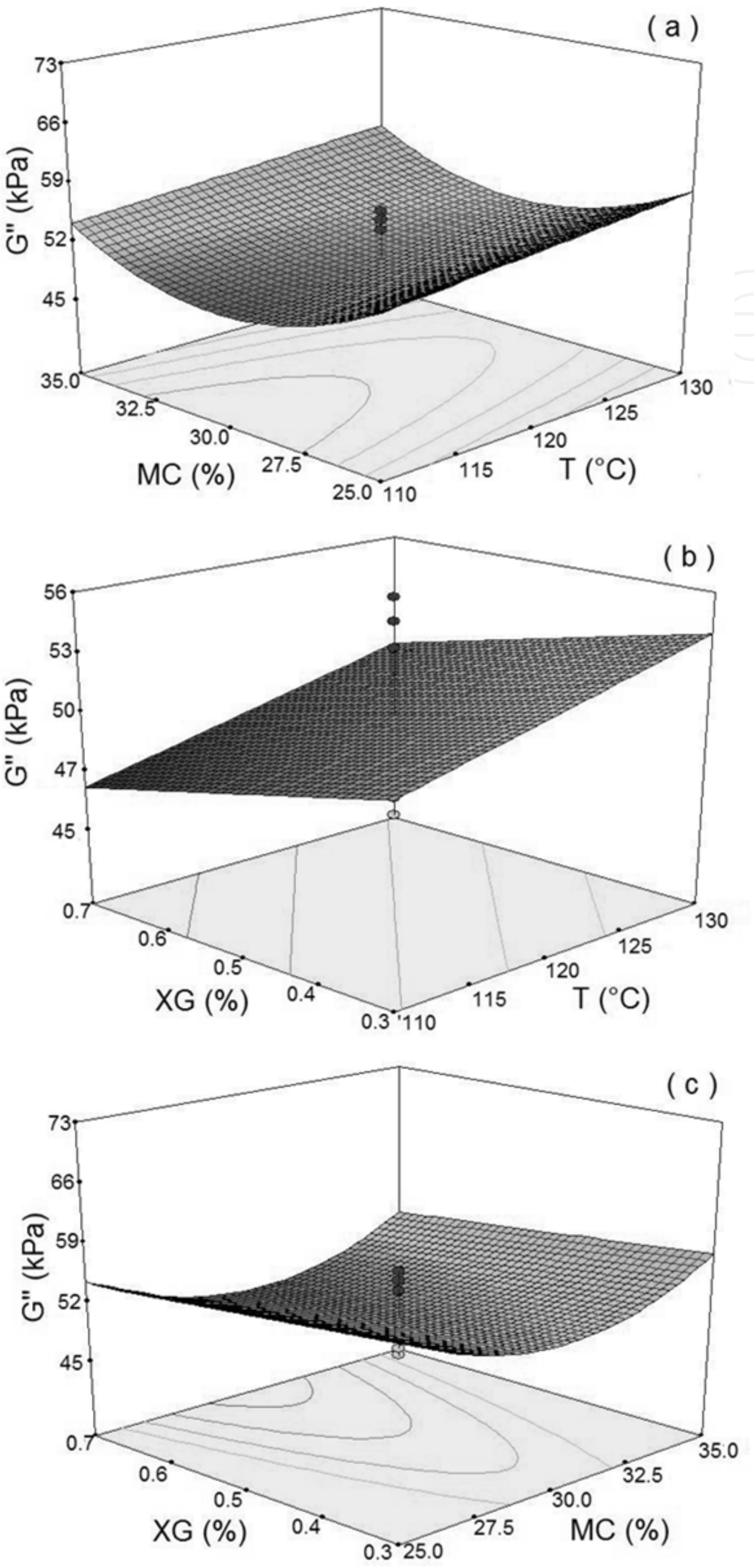


Figure 8. Response surface and contour plots for the effect of extrusion process factors on loss modulus (G'') of masa from extruded nixtamalized corn flour. (a) Effect of moisture content (MC, %) and extrusion temperature (T, °C) on G''; (b) Effect of xanthan gum (XG, %) and T on G''; (c) Effect of XG and MC on G''.

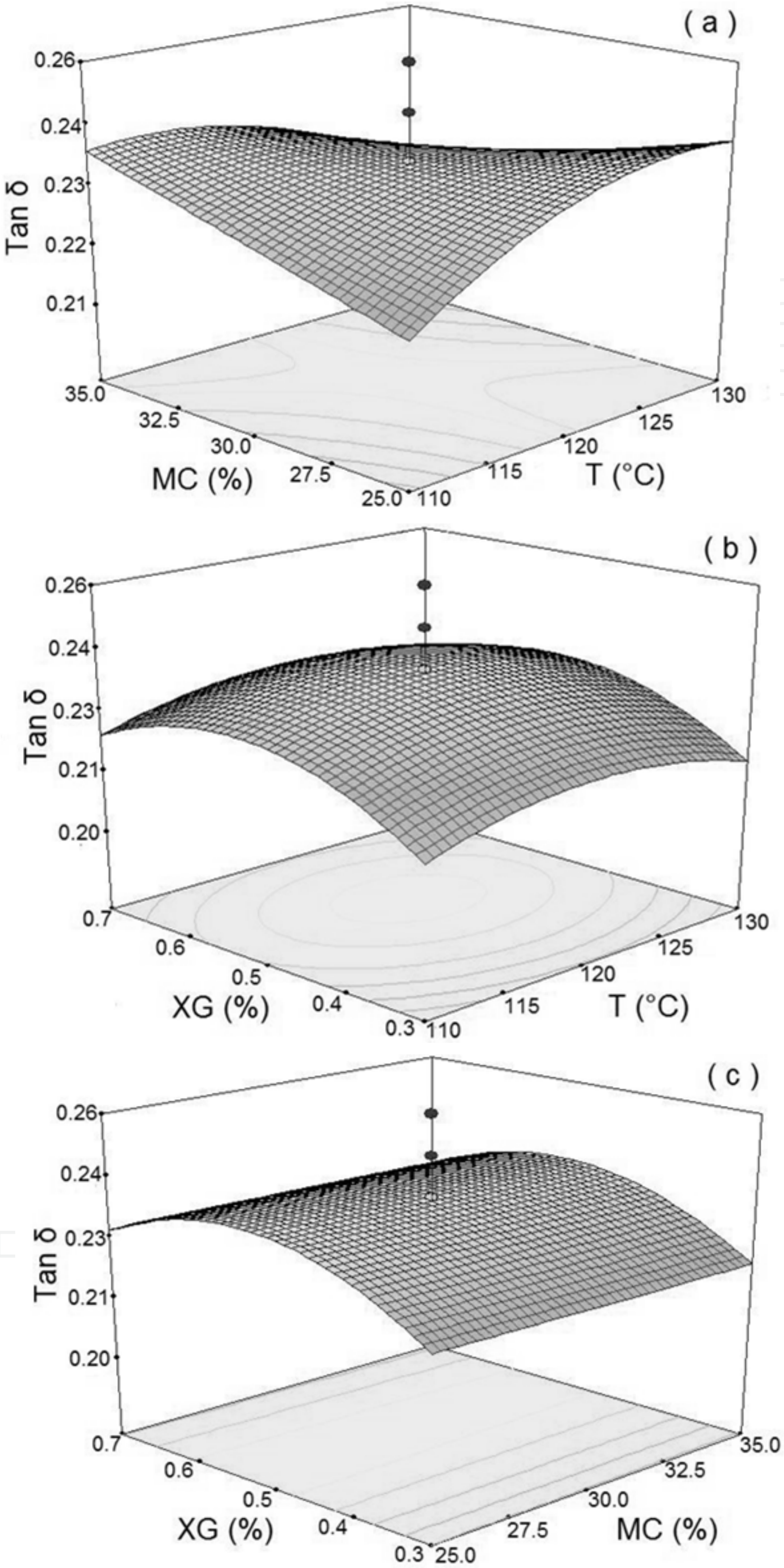


Figure 9. Response surface and contour plots for the effect of extrusion process factors on tangent of the phase angle (Tan δ) of masa from extruded nixtamalized corn flour. (a) Effect of moisture content (MC, %) and extrusion temperature (T, °C) on Tan δ; (b) Effect of xanthan gum (XG, %) and T on Tan δ; (c) Effect of XG and MC on Tan δ.

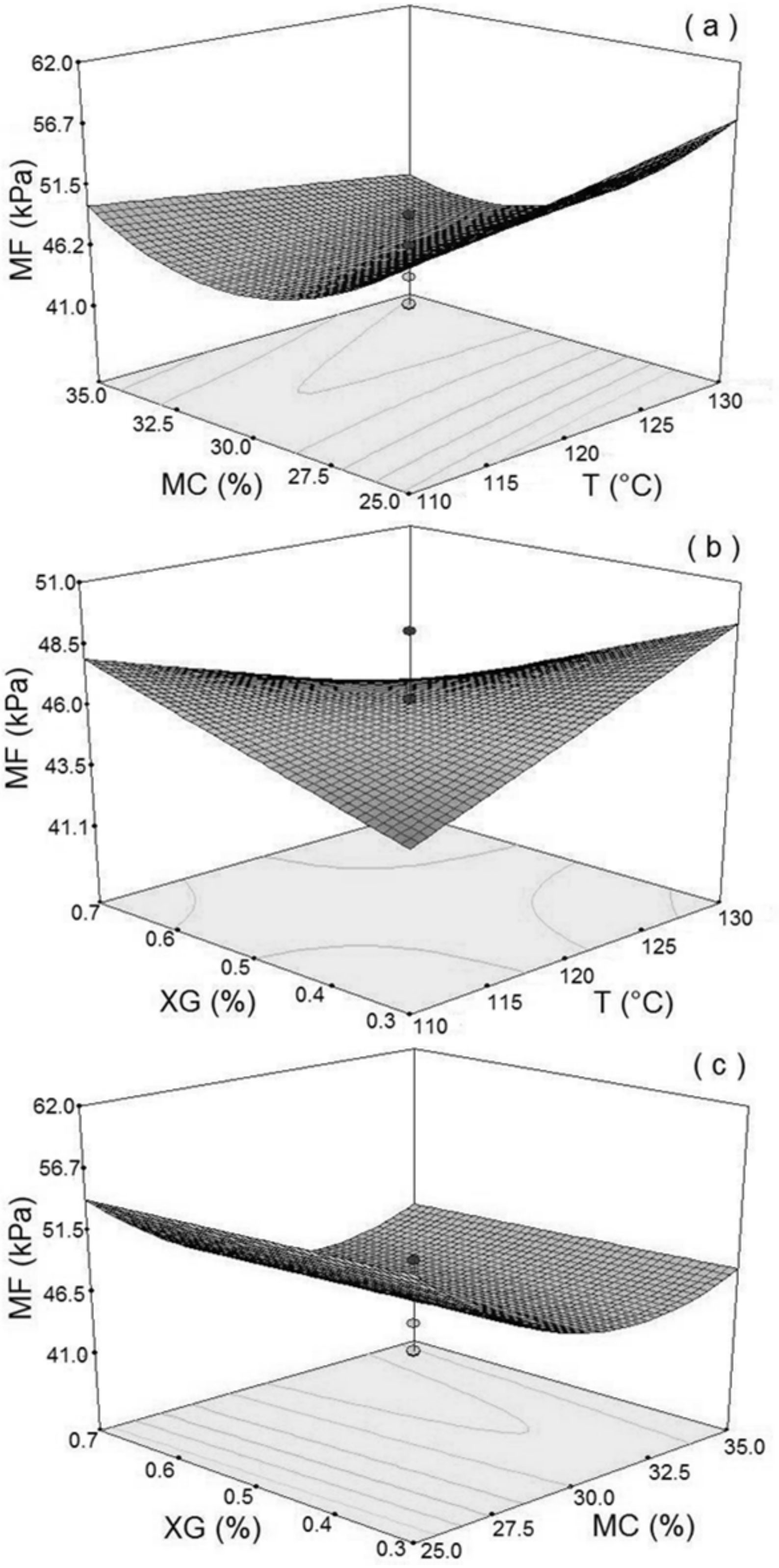


Figure 10. Response surface and contour plots for the effect of extrusion process factors on maximum force (MF) for tortilla made from extruded nixtamalized corn flour. (a) Effect of moisture content (MC, %) and extrusion temperature (T, °C) on MF; (b) Effect of xanthan gum (XG, %) and T on MF; (c) Effect of XG and MC on MF.

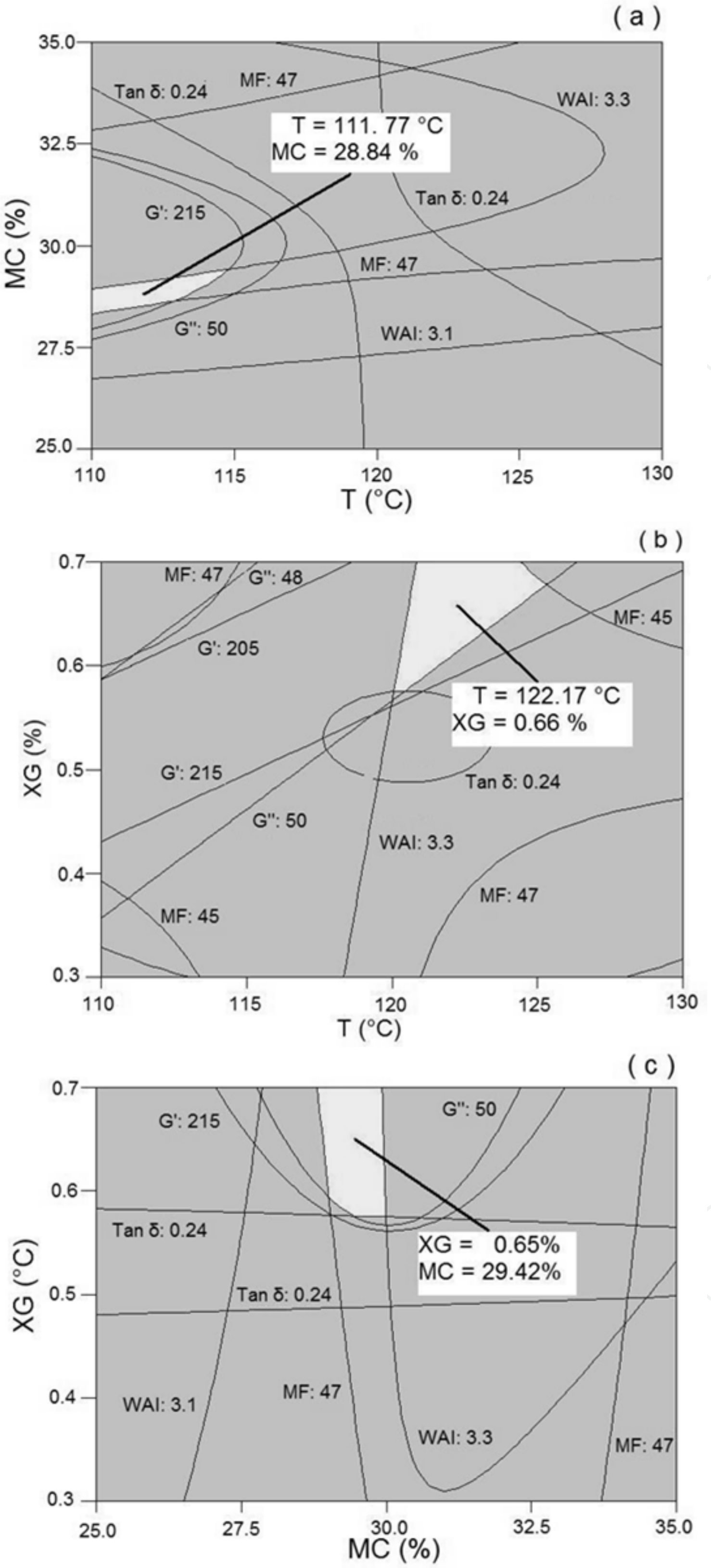


Figure 11. Regions of best combinations of process factors for producing optimized extruded nixtamalized corn flour, using a single-screw extruder. (a) Effect of moisture content (MC, %) and extrusion temperature (T, °C); (b) Effect of xanthan gum (XG, %) and T; (c) Effect of XG and MC.

Maximum force. Changes in MF of tortilla made from ENCFs were affected by the linear term of moisture content (MC, $p < 0.01$), quadratic of moisture content $(MC)^2$, $p < 0.01$) and the combinations of temperature-moisture content [(T)(MC), $p < 0.10$] and temperature-gum [(T)(XG), $p < 0.01$]. The prediction model in terms of original variables for MF was:

$$Y_{MF} = 14.75 - 7.36MC - 0.04(T)(MC) - 1.17(T)(XG) + 0.2(MC)^2 \tag{5}$$

The regression model explained 82% of the total variation ($p < 0.01$) of MF of tortilla made from ENCFs. Figs. 10(a,b,c) show the effect of MF in tortilla made from ENCFs as a function of T, MC and XG, observing that at any value of moisture content for conditioning starting at approximately the midpoint of the matrix, the maximum force is increased due to the quadratic effect. The lowest value (46.16 kPa) was shown at T=115 - 130°C/MC=30.69-34.87% (w/w)/GX=0.42-0.7% (w/w) (Figs. 10 (a,b,c).

The superimposition of contour plots of the effect of variables of the extrusion process (T, MC and XG) on WAI of flour, G', G'' and Tan δ of masa, and MF of tortilla made from ENCFs, was used to obtain Figs. 11 (a,b,c), which in turn was utilized to determine the best combinations of the extrusion process variables. The central points of the regions of optimization in Figs. 11 (a,b,c) correspond to the values of the process variables of T=111.77°C/MC=28.84%, XG=0.66%/T=122.17°C and XG=0.65%/MC=29.42%, respectively. The optimal combination for the operation conditions of the single-screw extruder derived from the averages of those values were: T=116.67°C/MC= 29.13%/XG = 0.65%. The optimal conditions were validated using experiments, which were similar to those values predicted by RSM. These values can be used to obtain ENCF with the highest WAI and WAC, and tortillas with less firmness (softer) and more flexibility (more rollable).

4. Conclusions

The viscoelastic parameters (G', G'', and Tan δ) were affected by xanthan gum concentration, inducing a weakening of the existing structure of masas from extruded nixtamalized corn flour. Tortillas from masas with high values of Tan δ improved their textural characteristics. The optimal combination of variables to obtain ENCF using a single-screw extruder was: T=116.67°C/MC= 29.13% / XG= 0.65%. The optimal conditions were validated experimentally, and results were similar to those values predicted by RSM. Besides the information of basic nature that can be obtained with the dynamic method, it can be considered a practical rheological tool capable of differentiating masa from extruded nixtamalized corn flours.

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