



Extrusion of flavored corn grits: Structural characteristics, volatile compounds retention and sensory acceptability

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ARTICLE INFO

Article history:

Received 14 January 2013

Received in revised form

14 June 2013

Accepted 25 June 2013

Keywords:

Flavor

Thermoplastic extrusion

Corn grits

Pre-flavored

Response surface methodology

ABSTRACT

The effects of the moisture content of the raw material, extrusion temperature and screw speed on flavor retention, sensory acceptability and structure of corn grits extrudates flavored with isovaleraldehyde, ethyl butyrate and butyric acid were investigated. Higher temperature resulted in more expanded extrudates with lower density and cutting force, while higher moisture content increased ethyl butyrate retention. The most acceptable extrudates were those obtained with low moisture content, under conditions of high extrusion temperature and high screw speed, or low screw speed and low extrusion temperature, whereas the aroma intensity closest to the ideal was observed under conditions of low extrusion temperature and low moisture content of the raw material.

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1. Introduction

Flavor is the sensory characteristic of food that is most affected in processes that use high temperatures, such as the thermoplastic extrusion. In the extrusion process, when the material leaves the die, expansion occurs and much of the volatiles are lost along with the steam (Reifsteck & Jeon, 2000; Yuliani, Bhandari, Rutgers, & D'Arcy, 2004).

Several factors are involved in volatile retention or loss during extrusion, including: raw material composition; extrusion conditions such as residence time, extruder temperature, moisture content of the raw material, compression and pressure; format and size of the final product; vapor loss during expansion; and diffusivity of the volatiles in the mass (Reifsteck & Jeon, 2000; Bhandari, D'Arcy, & Young, 2001; Yuliani et al., 2004).

One of the methods most commonly used for flavoring by the food industry is aromatization after extrusion, in which the flavor is sprayed onto the final product. This method, although greatly adding flavor to the extrudate, thereby increasing the pleasure sensation at the time of consumption, increases the fat content of

the product and may lead to nutritional imbalance when consumed in large quantities. The lipid content in extrudates that are flavored post-extrusion ranges from 18 to 41 g/100 g, with a caloric value of 450–575 calories per 100 g of product (Heyhoe, 2000).

However, new forms of flavoring have been studied in order to reduce the fat content and the caloric value of extrudates, including pre-extrusion flavoring. In this flavoring method, flavor is added to the raw material to be extruded, thus providing uniform distribution and better oxidative stability. This flavoring method is more suitable because no lipid vehicle is needed for it to be implemented. However, considerable loss of the volatile compounds added may occur during processing, with possible changes to the texture and structure of the extrudates (Bhandari et al., 2001).

Few studies relating to this topic are found in the literature (Yuliani, Torley, D'Arcy, Nicholson, & Bhandari, 2006a; Yuliani, Torley, D'Arcy, Nicholson, & Bhandari, 2006b; Yuliani, Torley, & Bhandari, 2009; Conti-Silva, Arêas, & Bastos, 2012). Thus, further work to develop better understanding of the effect of extrusion conditions on the structure and retention of flavor in pre-flavored extrudates is required.

Therefore, the aim of this study was to investigate the effects of the moisture content of the raw material, extrusion temperature and screw speed on the structural parameters, volatile compounds retention and sensory acceptability of corn grit extrudates flavored using response surface methodology.

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2. Materials and methods

2.1. Materials

The corn grits (7.7 g/100 g protein, 1.1 g/100 g fat, 0.3 g/100 g ash and 90.0 g/100 g total carbohydrates, on a dry basis) were purchased from a local market and were not subjected to any process before extrusion. For flavoring, a mixture of three volatile liquid compounds was used: isovaleraldehyde, ethyl butyrate and butyric acid (Sigma–Aldrich, Milwaukee, USA).

2.2. Corn grits composition

The corn grits composition was determined in accordance with the AOAC (1997) for ash and proteins, and in accordance with the AOCS (2009) specifications for lipid content, and the total carbohydrates content was estimated by difference. The corn grits were ground in a knife mill (model 340, Marconi, Piracicaba, Brazil) and the analyses were performed in triplicate.

2.3. Experimental design

The response surface methodology was applied using a rotational central composite design for three independent variables (Barros-Neto, Scarminio, & Bruns, 2010), namely: the moisture content of the raw material (dry basis), the extrusion temperature (temperature in third barrel zone) and the screw speed. The dependent variables used were the expansion ratio, density, cutting force and volatile retention for each compound individually and in total for all the compounds. Seventeen tests were performed: eight tests of factorial points (2^3) (three levels for each factor), six axial points (two for each variable) and three repetitions of the central point (Table 1).

The results from the dependent variables were subjected to multiple regression analysis using the Statistica 7.0 software (StatSoft Inc., Oklahoma, EUA) and coefficients with p values below 0.05 were considered significant. The regression was evaluated by means of analysis of variance: the regression was considered to be significant when $p \leq 0.05$, but no lack of fit at $p > 0.05$. Linear and quadratic models were tested to explain the influence of independent variables on the response variables, because in Response Surface Methodology, the relationship between these variables is

unknown and, therefore, it is necessary to find an adequate approximation to the true relationship between the response and the independent variables (Montgomery & Runger, 2006).

2.4. Adjustment of the moisture content of the corn grits

Samples of 400 g of grits were prepared to achieve moisture contents of 10, 12, 15, 18 and 20 g/100 g on a dry basis. The amount of water required to raise the moisture content of the corn grits to 15, 18 and 20 g/100 g (db) was added to the sample under constant stirring with the aid of a planetary mixer at low speed. After addition of water, the samples were packed in polyethylene bags and refrigerated for 24 h for homogenization. To adjust the moisture content of the samples to 10 and 12 g/100 g on a dry basis, drying was performed at 70 °C for approximately 60 and 30 min, respectively. The moisture content of the corn grits after adjustment to the desired values was then determined by drying at 105 °C (AOAC, 1997).

2.5. Flavoring of the corn grits

Each volatile compound was added at proportion of 1.5 g/100 g to the corn grits, as described by Conti-Silva et al. (2012). The volatiles were added by volume, based on the density of the compounds. Therefore, 7.53, 6.83 and 6.26 mL of isovaleraldehyde, ethyl butyrate and butyric acid, respectively, were added to 400 g of corn grits to each extrusion conditions. Sample homogenization was performed manually in the packaging and then the packages were sealed and kept at room temperature for 2 h before extrusion.

2.6. Extrusion process

The flavored corn grits were extruded in a single screw extruder (LAB 20, AX Plásticos, Diadema, Brazil) with four independent heating zones. The first and second zones were maintained at 50 and 90 °C, respectively; the third zone was adjusted according to the experimental design (Table 1); and the fourth zone was adjusted to 10 °C below the temperature of zone 3. The length/diameter ratio of the barrel was 26:1, and the screw used had a compression ratio of 4.6:1. The die diameter was 3.3 mm and feed rate was kept constant at 46 g min⁻¹.

Table 1

Experimental design with encoded and real values of the independent variables used under each extrusion condition.

Extrusion conditions	Encoded variables			Real variables		
	X_1	X_2	X_3	Moisture content (g/100 g)	Extrusion temperature (°C)	Screw speed (rpm)
1	−1	−1	−1	12	157	165
2	+1	−1	−1	18	157	165
3	−1	+1	−1	12	177	165
4	+1	+1	−1	18	177	165
5	−1	−1	+1	12	157	175
6	+1	−1	+1	18	157	175
7	−1	+1	+1	12	177	175
8	+1	+1	+1	18	177	175
9	−1.682	0	0	10	167	170
10	+1.682	0	0	20	167	170
11	0	−1.682	0	15	150	170
12	0	+1.682	0	15	184	170
13	0	0	−1.682	15	167	162
14	0	0	+1.682	15	167	178
15	0	0	0	15	167	170
16	0	0	0	15	167	170
17	0	0	0	15	167	170

2.7. Evaluation of the structural parameters of the extrudates

2.7.1. Expansion ratio

The expansion ratio was determined from 15 random measurements on the diameter of the extrudates using digital calipers (Digimess IP54), in accordance with the following equation: expansion ratio = mean diameter of the extrudates/die diameter.

2.7.2. Density

The density was determined from 15 random measurements on the diameter (D, cm) and length (L, cm) of the extrudates using digital calipers (Digimess IP54), and the weight (W, g) was determined on an analytical balance. The density (g cm^{-3}) was obtained from the following equation: $\rho = 4W/\pi D^2 L$ (Chávez-Jáuregui, Silva, & Arêas, 2000).

2.7.3. Cutting force

The force required to completely break the extrudates was determined using the TAXT2i equipment (Stable Micro Systems, Godalming, Inglaterra) and the “Texture Expert” software (Stable Micro Systems, Godalming, Inglaterra), using a probe with a knife blade set. Ten samples of approximately 5 cm in length were cut perpendicularly by the probe and the peak maximum force required was taken to be the cutting force of the extrudate.

2.8. Analysis on volatile compounds retention in the extrudates

Two grams of milled extrudate were added to vials (duplicates for each extrusion condition), and the volatile compounds present in the extrudates were captured using an automated headspace sampler (40 HStrap, Perkin Elmer, Shelton, USA). The following conditions were used: heating the vial at 70 °C for 30 min; needle temperature of 80 °C; vial pressurization time of 3 min; transfer line temperature of 210 °C; injection mode constant; injection duration of 0.1 min; injection pressure of 193 kPa; and column pressure of 159 kPa.

The compounds were then analyzed using a gas chromatograph (Clarus 680T, Perkin Elmer, Shelton, USA) coupled to a mass spectrometer (Clarus 600T, Perkin Elmer, Shelton, USA). A fused silica capillary column was used (Elite 5MS; 30 m $\times$ 0.25 mm $\times$ 1.4 μm , Perkin Elmer, Shelton, USA) with helium at a rate of 1 mL/min as carrier gas. The chromatographic conditions used were: injector at 230 °C; splitless mode until 1 min, split 1:100 until 1.5 min and split 1:200 until the end of the run; column programming starting at 40 °C for 3 min, with elevation to 210 °C at 25 °C min^{-1} , and remaining at 210 °C for 2 min (total run time 12 min). The mass spectrometer conditions were: interface temperature 230 °C; ionization source for electron impact at 70 eV and 210 °C; and extension of mass between 40 and 120 m/z .

The volatiles were injected separately at different known concentrations (four concentrations for each compound), in order to construct standard curves for each compound. The amount of each volatile compound retained in the extrudates was determined from the respective standard curve.

The chromatograms and spectra obtained were analyzed using the TurboMass software, version 5.4.2 (PerkinElmer Inc., Shelton, EUA).

2.9. Sensory analysis on extrudates

Sensory analysis was performed at the Sensory Analysis Laboratory, Department of Food Technology and Engineering, Instituto de Biociências, Letras e Ciências Exatas, Universidade Estadual Paulista “Julio de Mesquita Filho”, using individual booths with white light. This study was approved by the Research Ethics Committee of the same institute (Opinion 050/11).

The panelists received 4.5 g of the extrudates in plastic cups coded with three digits and covered with two layers of aluminum foil: the first with orifices for suction of flavor and the second without orifices to prevent loss of volatile compounds. The sensory analysis was performed in two sessions: nine samples were evaluated in the first session and seven in the second one. The samples were presented in the form of complete random blocks so balanced and monadic.

Ninety untrained panelists were recruited in the first session of the test, but only sixty six panelists returned to finish the test. Therefore, the sensory panel was formed by sixty six panelists.

They were asked to give their opinions regarding the acceptability of the product aroma. Two scales were used: 1) hedonic scale of 9 points (9 = extremely liked; 5 = neither liked nor disliked; 1 = extremely disliked), to assess how much the panelists liked the flavor of the products; and 2) a just-about-right (JAR) scale of nine points (9 = extreme of higher intensity than ideal, 5 = ideal intensity, 1 = extreme of lower intensity than ideal), to assess how perfect the intensity of the flavor products was (Meilgaard, Civille, & Carr, 1999).

The results from the JAR scale were adjusted in accordance with Bower and Boyd (2003). For this, the ideal flavor intensity was taken to be the dependent variable.

2.10. Correlation analysis

Pearson correlation analysis was performed between the structural parameters and between the amount of volatile compounds and the sensory acceptability of the extrudates using the PASW Statistics 18 software (SPSS Inc., Hong Kong, China).

3. Results and discussion

3.1. Structural parameters

The expansion ratio of the extrudates ranged from 1.61 to 3.08, which was considered to be good expansion, given that addition of volatile components prior to extrusion can reduce the extrudate expansion. These expansion ratio values were similar to those found by Conti-Silva et al. (2012), who observed expansion ratios of 2.9–3.7 for extruded corn grits flavored with the same volatile compounds used in this study, and higher than those found by Yuliani et al. (2009), who obtained expansion ratios of 1.7–2.2 for extrusion of corn starch aromatized with encapsulated D-limonene.

The best fit to the expansion ratio was observed for the linear model, and only the extrusion temperature was significant (Table 2). The increase of extrusion temperature enhanced the expansion ratio of the extrudates (Fig. 1), which can also be observed by the positive sign of the coefficient of the linear term of temperature in Table 2. This effect was due to increasing size of the air cells caused by steam conduction. When the dough left the die, the sudden drop in pressure caused rapid evaporation of the superheated water present in the material. This led to formation of bubbles, which grew in mass due to the pressure difference between the mass and the atmospheric pressure, thereby resulting in the expansion of the final product. The higher the extrusion temperature was, the lower the viscosity of the dough and the higher the temperature of the superheated water present in the dough were, thus increasing the pressure differential at the exit from the extruder and promoting formation of bubbles and expansion of the material (Campanella, Li, Ross, & Okos, 2002). Saeleaw, Dürschmid, and Schleining (2012) and Yu, Ramaswamy, and Boye (2013) observed the same behavior in relation to the expansion ratio of rye flour extrudates and extrudates prepared from blends soy protein isolate and corn flour, respectively.

Table 2
Models and goodness of fit for the dependent variables.

Dependent variable	Equation	R ² (%)	p-value	Lack of fit
Expansion ratio (ER)	$Y_{ER} = 2.508 + 0.356T$	50.43	0.024	0.385
Density (D)	$Y_D = 0.369 - 0.174T$	60.07	0.006	0.251
Cutting force (CF)	$Y_{CF} = 37.905 - 8.525T$	61.97	0.005	0.618
Ethyl butyrate retention (EBR)	$Y_{EBR} = 0.204 + 0.058M$	38.67	0.086	0.710
Acceptability – hedonic scale (HS)	$Y_{HS} = 5.325 - 0.230M + 0.114T \cdot S$	89.83	0.009	0.299
Acceptability – adjusted JAR scale (A-JAR-S)	$Y_{A-JAR-S} = 6.6 - 0.199M + 0.186M \cdot T$	77.96	0.098	0.431

T = extrusion temperature, M = moisture content of raw material, S = screw speed.

The density of the extrudates ranged from 0.13 to 0.85 g cm⁻³ and was below the values found by Yuliani et al. (2006a) and (2006b) from extrusion of corn starch aromatized with encapsulated D-limonene, and Yuliani et al. (2009) from extrusion of corn starch flavored with unencapsulated D-limonene. Moreover, Conti-Silva et al. (2012) found density values of 0.12–0.28 g cm⁻³ for extrusion of flavored corn grits, i.e. lower density values than were found in the present study.

The best fit to the density of the extrudates was observed for the linear model, and only the extrusion temperature was significant (Table 2). Increasing the temperature reduced the density of the extrudates, i.e. the effect was the inverse of what was found for the expansion ratio, which can be observed by the negative sign of the coefficient of the linear term of temperature (Table 2). This relationship was also observed through the Pearson correlation coefficient between the expansion ratio and density ($r = -0.952$, $p < 0.001$), thus indicating a strong negative correlation between these two dependent variables. Density is a parameter that can also be used to assess the degree of expansion of the extrudates. While the expansion ratio considers only the cross-section of the material, density considers expansion in all directions. Low density is desirable for extruded products (Meng, Threinem, Hansen, & Driedger, 2010). The same temperature effect on extrudate density was observed by Yuliani et al. (2009) in relation to extrusion of corn starch with D-limonene and by Saeleaw et al. (2012) in relation to extrusion of rye flour.

The cutting force of the extrudates ranged from 20.98 to 51.60 N, which was close to the range of values found by Conti-Silva et al. (2012) for the cutting force of flavored corn grit extrudates, which was 23.7–34.2. The best fit for the cutting force of extrudates was also observed for the linear model, and only the extrusion temperature was significant (Table 2).

It was observed that increasing the extrusion temperature not only decreased the density but also decreased the cutting force of the extrudates, also verified by the negative sign of the coefficient of the linear term of temperature (Table 2). Since temperature

increases reduce the viscosity of the dough and promote growth of air bubbles, the thickness of cell walls in the extrudates decrease (Yuliani et al. 2006a), thus reducing the cutting force.

The cutting force of the extrudates was negatively correlated with the expansion ratio ($r = -0.628$, $p = 0.007$) and positively correlated with the density ($r = 0.726$, $p = 0.001$), given that extrudates presenting greater expansion or lower density may be structurally more fragile or have lower mechanical strength (Yuliani et al., 2009).

3.2. Volatile compounds retention

Volatile compounds retention ranged from not-detected (ND) to 0.49 mg/g of extrudate for isovaleraldehyde, from 0.05 to 0.62 mg/g of extrudate for ethyl butyrate and from ND to 36.10 mg/g of extrudate for butyric acid. The bigger retention was found to the butyric acid, followed by ethyl butyrate and isovaleraldehyde, as found by Conti-Silva et al. (2012). This behavior is due to vapor pressure and boiling temperature of the volatile compounds. Isovaleraldehyde, the compound less retained in all extrusion conditions, has the biggest vapor pressure (4009 Pa) and lowest boiling temperature (92.5 °C), as opposed to butyric acid that was more retained because of the lowest vapor pressure (57 Pa) and biggest boiling temperature (163.7 °C) (Lide, 1997). The low volatility promotes a higher diffusivity of the compound through the matrix of the extrudate, resulting in a bigger encapsulation and, consequently, higher retention. Moreover, the hydrophilic polarity of the starch favors retention of polar flavor molecules, as butyric acid, due to the capacity of the starch to form hydrogen bonds with aroma compounds (Boutboul, Giampaoli, Feigenbaum, & Ducruet, 2002).

The best fit for retention of ethyl butyrate was observed for the linear model ($p < 1$) and only the moisture content of the raw material was significant. Higher moisture content of the corn grits increased the retention of this compound, which can be verified by the positive sign of the coefficient of the linear term of moisture content (Table 2). Conti-Silva et al. (2012) observed greater retention of ethyl butyrate in corn grit extrudates under extrusion conditions that were less severe (20 g/100 g moisture and extrusion temperature 90 °C) than those used in the present study. However, none of the previous studies that evaluated the effect of extrusion conditions on flavor retention in extrudates using the response surface methodology (Yuliani et al., 2009; Yuliani et al., 2006a, 2006b) studied the effect of moisture content of the raw material on this retention. Therefore, the results found in the present study could not be compared with those of other authors.

The adjusted models to retention of isovaleraldehyde and of butyric acid were not significant.

3.3. Sensory acceptability

The means for flavor acceptability on the hedonic scale ranged from 4.88 to 5.92, i.e. between “disliked slightly” and “liked slightly”. The acceptability of the extrudate flavor on the hedonic

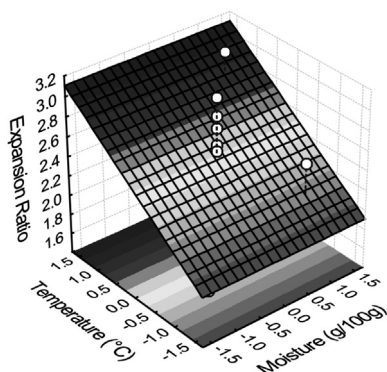


Fig. 1. Expansion ratio of extrudates as a function of the temperature and moisture content of the raw material, with screw speed of 165 rpm.

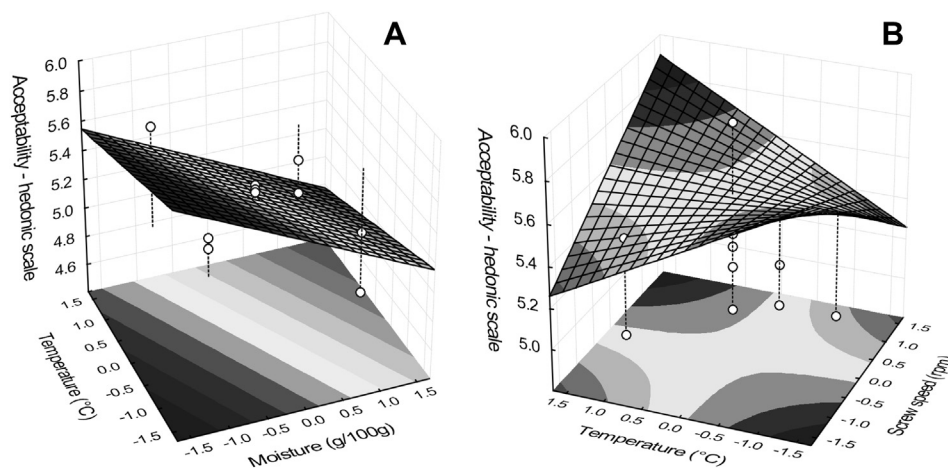


Fig. 2. Acceptability of flavor on the hedonic scale as a function of temperature and moisture content of the raw material, with screw speed of 165 rpm (A), and as a function of screw speed and temperature, with moisture content of the raw material of 12 g/100 g (B).

scale was dependent of the linear term of the moisture content of the raw materials and also of the interaction between extrusion temperature and screw speed (Table 2). The reduction in moisture content of the raw material resulted in increased acceptability of the extrudate flavor among the panelists (Fig. 2A). Greater acceptability of extrudate flavor was also observed with increasing screw speed at high temperature and decreasing temperature of extrusion at low screw speeds (Fig. 2B).

There was a strong negative correlation between the amount of volatile flavor and acceptability on the hedonic scale ($r = -0.759$, $p < 0.001$ for isovaleraldehyde; and $r = -0.785$, $p < 0.001$ for ethyl butyrate), even when the quantities of the three volatiles were summed ($r = -0.772$, $p < 0.001$). This shows that when the volatiles were present in minor amounts, the acceptability of the extrudates was higher, thus indicating that lower volatile retention after extrusion was a contributory factor toward the acceptability of the products.

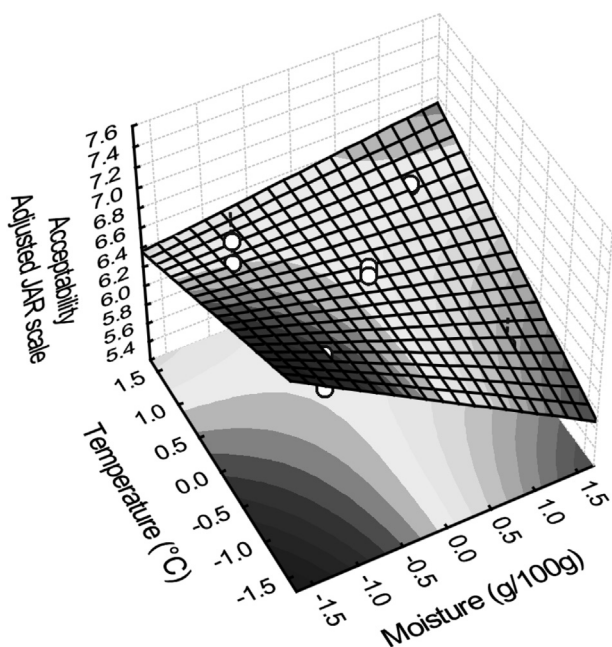


Fig. 3. Acceptability of flavor on the adjusted JAR scale as a function of temperature and moisture content of the raw material, with screw speed of 165 rpm.

On the adjusted JAR scale, the value of 9 indicated the “ideal intensity” for the characteristic evaluated, and the further away from 9 that this value was, the less ideal the intensity this characteristic was, independent of whether it was more or less intense than the ideal (Bower & Boyd, 2003). The ideal values adjusted for the flavor intensity ranged from 5.73 to 7.23. The acceptability of the flavor intensity of the extrudates on the adjusted JAR scale was dependent of the linear term of the moisture content of the raw material and the interaction of the moisture content of the raw material with the extrusion temperature (Table 2). Increasing the extrusion temperature at high moisture content resulted in extrudates with flavor intensity close to the ideal. However, extrudates with flavor intensity values closer to the ideal were observed with decreasing temperature at low moisture content (Fig. 3).

The specific mechanical energy is a measure of the work done by the extruder on the material and results of process conditions, such as moisture of the material. The water can act as a lubricating agent during processing, favoring the flow and reducing shearing of the material inside extruder (Campanella et al. 2002; Kokini, Chang, & Lai, 1992). Therefore, when moisture is reduced, acceptability by adjusted JAR scale increases, probably because of shear increasing and consequent decrease of volatile compounds retention (Fig. 2).

4. Conclusion

Increasing the extrusion temperature resulted in extrudates with greater expansion, lower density and lower cutting force, while the retention of ethyl butyrate in the extrudates increased with increasing moisture content of the raw material.

The flavor acceptability on the hedonic scale was dependent of the moisture content of the raw material and of the interaction between extrusion temperature and screw speed. The most acceptable extrudates were processed with lower moisture, under conditions of high extrusion temperature and high screw speed, or low screw speed and low extrusion temperature.

The flavor acceptability intensity on the adjusted JAR scale was influenced by the moisture content of the raw material and the extrusion temperature. Flavor intensity closer to the ideal was observed at low extrusion temperature and low moisture content of the raw material.

Among the extrusion conditions studied for extruding flavored corn grits, those using elevated temperature favored extrudate expansion, while low moisture content of the raw material favored

sensory acceptability of the flavor due to lower retention of ethyl butyrate in the final product.

Acknowledgments

The authors are grateful for financial support from CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnológico), FAPESP (Fundação de Amparo à Pesquisa do Estado de São Paulo) (grant 2010/09998-6) and the Pro-Rector for Research of UNESP (Universidade Estadual Paulista).

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