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Effect of some operational extrusion parameters on the constituents of orange pulp

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Abstract

Orange pulp was extruded using a Brabender laboratory single screw extruder (20:1 L/D), with the objective of modifying the properties of the fibre components. Response surface methodology was used following a central rotational composite, experimental design (5^3). The independent variables studied were: barrel temperatures (83, 100, 125, 150, and 167 °C); moisture contents (22%, 25%, 30%, 35% and 38%) and screw speed (126, 140, 160, 180 and 190 rpm). The feed speed was kept constant (70 g/min). The compression ratio was 3:1, and the diameter of the die 4 mm. The extrusion process decreased insoluble dietary fibre in 39.06% and soluble dietary fibre was increased by 80%. Also, total dietary fibre was decreased as consequence of the production of smaller fragments that would not be completely recovered during the alcoholic precipitation. Increments in contents of total pectin and soluble pectin resulted from the solubilization of pectic substances.

Keywords: Extrusion; Pectin; Orange pulp; Dietary fibre

1. Introduction

The use of by-products derived from the citric industry and principally staple fibers contained in the membranes, juice vesicles, albedo, flavedo, seeds, and rinds of oranges can be increased improving their functional properties (Artz, Warren, & Villota, 1990).

Most of the worldwide orange production is concentrated in Brazil (approximately 50%), USA (30%) and the rest (20%) is supplied by other countries (Strohl, 1981).

Individuals attain a stable of well being after the ingestion of foods containing dietary fiber their formulations, which is therefore, also recommended in the reduction of weight and obesity (Gordon, 1989). Feeding of rats with a 15% orange pulp diet indicate a clear tumor suppressor effect of these dietary fibers (Kossoy, Ben-Hur, Stark, Zusman, & Madar, 2001).

In the stomach, the dietary fiber acts as a thickening agent, increasing the volume of ingested foods and the time of gastric performance. Pectin, the main component of the soluble dietary fiber, increases the viscosity of the gastric contents, delaying the emptying stomach and reducing the proportion of absorption of the carbohydrates in the small intestine, thus reducing the level of seric glucose (Scheneeman, 1986).

Dietary fibre acts as a bulking agent, normalizing intestinal motility and preventing diverticular disease. Some types may also be important in reducing colonic cancer, in lowering serum cholesterol levels and in preventing hyperglycemia in diabetic patients. In recent years diverse products with high fibre content have been developed.

Various researchers have shown the potential use of fibre in breadmaking (Belshaw, 1978; Dubois, 1978; Gould et al., 1989; Gould & Freer, 1984; Ribeiro, 1996; Sasaki, Tanaka, Nambu, Sato, & Kainuma, 1979), meat

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products, dehydrated gravies, puddings, fed and extruded snacks (Camire & King, 1991; Kesterson & Braddock, 1973; Lanza, Priolo, Biondi, Bella, & Salem, 2001).

Also dehydrated juice vesicles could be used in the preparation of drinks in breadmaking and in the meat industry (Braddock, 1983; Fox, 1980).

The objective of this work was to evaluate the effect of some operational extrusion parameters on the constituents of orange pulp.

2. Materials and methods

Raw material. Washed orange pulp (90% moisture content) was obtained from Citrosuco Paulista S.A. Limeira, SP, Brazil. The samples were dried to about 9.0% moisture content in a forced-air oven at 80 °C for 12 h (Aguirre & Travaglini, 1987). The samples were milled using a Brabender Quadrumat Senior Mill (Duisburg, Germany) to produce particles with a size of <1.190 mm and stored in polyethylene bags for further analysis.

2.1. Extrusion process

Extrusion experiments were performed using a laboratory single-screw extruder (model GNF 1014/2, Brabender, Inc., Duisburg Germany) with an extruder barrel of 380 mm in length and 19 mm of diameter. The barrel had three sections with electric heaters independently controlled and a 3:1 compression ratio screw. A die head with a diameter of 4 mm was used. Compressed air was circulated around the barrel to maintain a precise control of the temperature of the barrel and die assembles. Flour samples were fed by forced feeding. The feed rate was maintained constant at 70 g/min (dry matter). The extrusion conditions were: barrel temperature 83, 100, 125, 150, and 167 °C; moisture content, 22%, 25%, 30%, 35 and 38%; and screw speed 126, 140, 160, 180 and 194 rpm. Samples were dried in a forced-air oven at 60 °C for 12 h (Artz et al., 1990) and milled using a roller mill (Brabender Inc., Duisburg Germany) to produce particles with a size of <0.840 mm and stored in polyethylene bags for further analysis.

2.2. Chemical analysis

Official methods (AACC, 1995) were used to analyze moisture (no. 44-15A), protein (no. 46-13), lipid (no. 30-10) and ash (no. 08-01) contents. The enzymatic–gravimetric method of (Prosky, Asp, Furda, Devries, & Schweeizer, 1988) was used to determine soluble dietary fibre (SDF) and insoluble dietary fibre (IDF). Dehydrated samples were hydrolysed using thermoresistent α-amylase (A-3306 Sigma), protease (P-3910 Sigma) and amyloglicosidase (A-9913 Sigma), for the removal of protein and starch. After enzymatic hydrolysis, the IDF was filtered and separated, and the SDF precipitated using 4 volumes of ethanol (98%). The alcoholic solution was then filtered and the residues and precipitates were washed with ethanol (78%), ethanol (95%) and acetone, dried and weighed. Due to the presence of protein and ash residues, the values of IDF and SDF were corrected. Total dietary fibre (TDF) was calculated from the sum of IDF and SDF (Gourgue, Champ, Lozano, & Delort-Laval, 1992). Total sugars were determined according to the orcinol-sulphuric acid method (Tollier & Robin, 1979). The standard curve was prepared with glucose at 420 nm. The uronic acids (expressed as anhydric galacturonic acid) were extracted and analysed (Ahmed & Lavavitch, 1977); and the concentrations used established (Blumenkrantz & Asboe-Hansen, 1973). All the analyses were determined in duplicate.

2.3. Particle size distribution of the orange pulp

Particle size distribution was determined with 30 g of sample using the Produtest equipped with sieves 20, 32, 35, 60, 80 100 mesh plus the base and a shaking time of 15 min (Henderson & Perry, 1976) using the following equation:

$$D = 104.14 \times 2^{MF}$$

where, D is the mean particle diameter (μm), and FM is the fitness modulus (percentage of the weight of the fractions retained by each sieve).

The particle size distribution of orange pulp showed that 62.88% of the particles were retained by sieves of 32, 35 and 60 mesh, with an average size from 250 to 840 μm. Also, 35.16% of the particles were of smaller size (<250 μm) being retained by the sieve 80 and the base, and only 2.80% were retained by sieve 20 representing the oversize particles (>840 μm).

2.4. Experimental design and data analysis

Response Surface Methodology (RSM) was chosen to build up some mathematical models, using a central composite rotational design, making it possible to quantitatively interpret and describe the relationship between the selected dependent extrusion variables and the extrusion parameters. Such models have always proved to be very useful for the analysis and prediction process. A central rotational composite, experimental design, with a $\propto = 1.682$ (Table 1) according to the theory and rules of Box and Wilson (1951). The outline of the experimental design and their independent variables and variation levels are presented in Table 2.

The data were analysed using Statistic 6.0 for Windows program (SAS, 1992) to investigate the trends

Table 1
Coded levels for independent variables

Variable	Level of variation				
	$-\alpha = -1.682$	−1	0	+1	$+\alpha = +1.682$
X_1	83	100	125	150	167
X_2	21.6	25	30	35	38.4
X_3	126.38	140	160	180	193.62

X_1 = Temperature (°C); X_2 = Moisture content (%); X_3 = Screw speed (rpm).

Table 2
Independent variables and experimental design levels expressed in coded and natural units for extrusion

Treatment	Coded units		
	X_1	X_2	X_3
1	−1	−1	−1
2	+1	−1	−1
3	−1	+1	−1
4	−1	+1	+1
5	−1	−1	+1
6	+1	−1	+1
7	−1	+1	+1
8	+1	+1	+1
9	0	0	0
10	0	0	0
11	0	0	0
12	0	0	0
13	0	0	0
14	0	0	0
15	−1.682	0	0
16	+1.682	0	0
17	0	−1.682	0
18	0	+1.682	0
19	0	0	−1.682
20	0	0	+1.682

X_1 = Temperature (°C); X_2 = Moisture contents (%); X_3 = Screw speed (rpm).

insoluble dietary fibre, soluble dietary fibre, total dietary fibre, total pectin and soluble pectin were determined. It was assumed that one mathematical function is present for the response variable in terms of two independent processing factors.

3. Results and discussion

3.1. Chemical analysis of the orange pulp

The possibilities and potential use of orange pulp for industrial proposes and other uses are basically related to its dietary fibre content. Considerable attention had been focused on the incidence of a number of "non-infection diseases of civilization" attributed to a low dietary fibre intake.

The orange pulp analysed had 9.79% of proteins, 2.43% of lipids, 2.66% of ash contents, 9.27% of total carbohydrates and a high TDF content (74.87%), consisting of significant contents of both IDF (54.81%) and SDF (20.06%). Ribeiro (1996) reported similar results in the composition of fibre from orange pulp dried using a drum dryer.

The contents of protein, ash, lipids and carbohydrate (determined in the alcoholic extract) were similar to those previously reported (Areas, 1994; Fox, 1980; Grigelmo & Martin-Belloso, 1998; Kesterson & Braddock, 1973; Ribeiro, 1996), although Aguirre and Travaglini (1987) reported lower values for total carbohydrates (6.39%) in orange pulp dried using pneumatic equipment. The differences in the composition of fibre, found by these researchers are attributed to variations in the method of analysis, variety, maturation and edible fragments of the dietary source, as well as the method of drying used. Eaks and Sinclair (1980) reported that the contents of protein, ash and lipids were lower in mature oranges than in green oranges. Grigelmo and Martin-Belloso (1998) reported values of 35.4–36.9% for TDF, 15.7–16.3% for pectins, 16.6–18.1% for hemicellulose and 2.2–3.0% for lignin from orange pulp for three varieties of oranges. Also chemical analyses of orange TDF concentrate showed low contents of protein, fat and ash (8.1–10.1%, 1.5–3.0% and 2.6–3.1% DM, respectively).

3.2. Composition of the dietary fibre of orange pulp

Neutral detergent fibre (NDF) and acid detergent fibre (ADF) values were 33.44 and 28.09, respectively, similar values to those reported by Areas (1994). However, lower values for ADF (23.1%) were found in the edible portion of orange by McConnell, Eastwood, and Mitchell (1974). The value for hemicelluloses found in this study (5.35%) agrees with the results reported by Areas (1994); Eaks and Sinclair (1980). However, Braddock and Graumlich (1981) reported higher values for ADF (10.63%). These researchers did not specify if the determination was determined in washed orange pulp or not. Probably the successive operations of washing are the causes of the differences observed.

The values for cellulose (25.32%) and lignin (2.77%) were similar to those previously reported (Areas, 1994; Braddock & Graumlich, 1981; McConnell et al., 1974). However, Eaks and Sinclair (1980) considering the addition of cellulose and lignin determined in the rinds of

Table 3
Effects of the extrusion variables on dietary fibre and pectin content of orange pulp

Assays	Independent variables			Responses				
	X_1	X_2	X_3	IDF (%)	SDF (%)	TDF (%)	TP (%)	SP (%)
01	100	25	140	40.54	28.28	68.82	42.69	04.50
02	150	25	140	33.57	33.74	67.31	39.70	14.87
03	100	35	140	38.76	30.27	69.03	40.57	04.73
04	150	35	140	32.70	37.88	70.58	40.60	12.05
05	100	25	180	37.60	32.71	70.31	36.90	04.17
06	150	25	180	33.37	34.51	67.88	36.48	12.45
07	100	35	180	38.27	33.95	72.22	35.47	04.45
08	150	35	180	34.92	35.42	70.34	38.65	10.41
09	125	30	160	35.70	35.94	71.64	40.81	09.02
10	125	30	160	36.36	33.87	70.23	42.30	10.35
11	125	30	160	35.64	35.65	71.29	42.46	10.39
12	125	30	160	35.60	35.19	70.79	39.21	11.26
13	125	30	160	36.33	34.80	71.13	43.70	09.63
14	125	30	160	35.19	33.03	68.22	41.14	08.21
15	83	30	160	38.29	30.25	68.54	42.88	04.23
16	167	30	160	32.28	34.25	66.53	41.39	16.76
17	125	22	160	34.88	35.08	69.96	38.94	13.27
18	125	38	160	36.10	36.30	72.40	36.47	08.54
19	125	30	126	35.93	36.10	72.03	43.16	11.72
20	125	30	194	35.77	34.82	70.59	40.87	12.46
Raw orange pulp				54.81	20.06	74.87	35.45	02.07

X_1 = Barrel temperature (°C); X_2 = Feed moisture; (%); X_3 = Screw speed (rpm); IDF = Insoluble dietary fibre; SDF = Soluble dietary fibre; TDF = Total dietary fibre; TP = Total pectin; SP = Soluble pectin.

mature oranges of the variety Valence, reported values of 17.5%, while higher values were found for the same authors in green oranges (21.4%). Also, other researchers (Areas, 1994; Braddock & Graumlich, 1981; Fox, 1980) reported values of 32.84%, 30.0% and 38.9%, respectively for total pectin in orange pulp. Comparing these results with those found in this study, the pectic substance contents were 35.45% of the total, thus, indicating similarity with these results.

3.3. Effects of the extrusion variables on the composition of dietary fibre and pectin content of the orange pulp

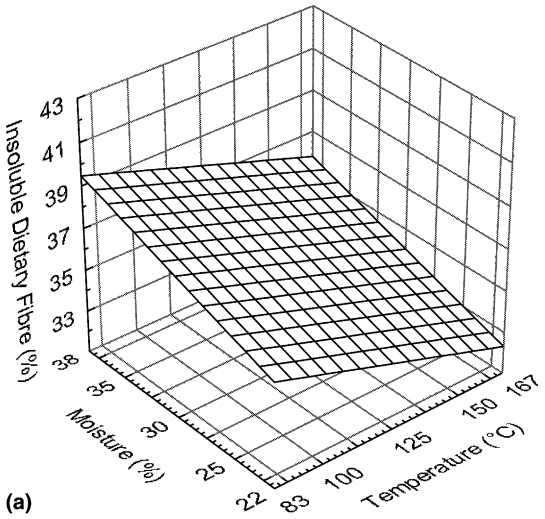
Table 3 shows the experimental results for the contents of IDF, SDF, TDF, total pectin (TP) and soluble pectin (SP) in each of the assays of the extrusion process with the orange pulp.

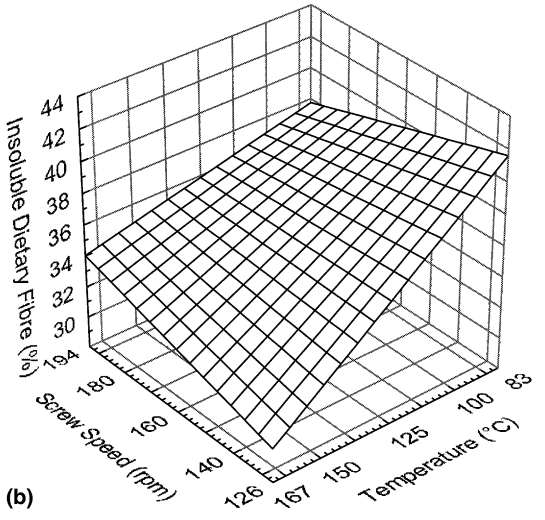
3.4. Insoluble dietary fibre and soluble dietary fibre

Fig. 1(a)–(c) show the fitted models of the variation of IDF content as a function of the independent variables. The IDF content decreased with higher barrel temperatures and lower moisture contents, when the screw speed was at the highest value (194 rpm).

However when the moisture content was fixed at the central point (30%), the lowest values for IDF were attained with higher barrel temperatures (higher than 150 °C) and longer residence times, with lower screw speed values (lower than 140 rpm) (Fig. 1(b)). When the barrel temperature was fixed at 125 °C, the lowest values for IDF were attained with longer times of residence (lower screw speed) and higher moisture contents (Fig. 1(c)). Probably under these conditions the residence time is adequate to assure the solubilization of the fibre components due to the effect of barrel temperature. The same behaviour was evidenced with lower moisture contents and higher screw speeds. The values obtained for IAF under each of the extrusion conditions (32.28–40.54%) were lower than those for raw orange pulp (54.81%). These results were expected, since previous studies (Björck, Nyman, & Asp, 1984; Ralet, Valle, & Thibault, 1993) reported the occurrence of a redistribution of the IDF as SDF during the extrusion process, causing reductions in the IDF levels. Similar behaviour was reported with wheat flour and bran (Wang, Klopfenstein, & Ponte, 1993) and blends of wheat and barley (Fornal, Soral–Smietana, & Szpenelowski, 1987). This effect would be the result of the breakage of covalent and non-covalent linkages between carbohydrates and proteins associated with the fibre, resulting in small molecular fragments, which would be more soluble. Gourgue, Champ, Guillon, and Delort-Laval (1994) reported that with extruded orange and lemon rinds the IDF contents decreased from 54.00% to 39.17% for orange, and from 47.94% to 33.66% for lemon.

Fig. 2 shows the mathematical model for SDF as a function of the barrel temperature (X_1) and screw speed (X_3) fixed at the central point of 30% moisture content. The highest values for SDF were obtained when the





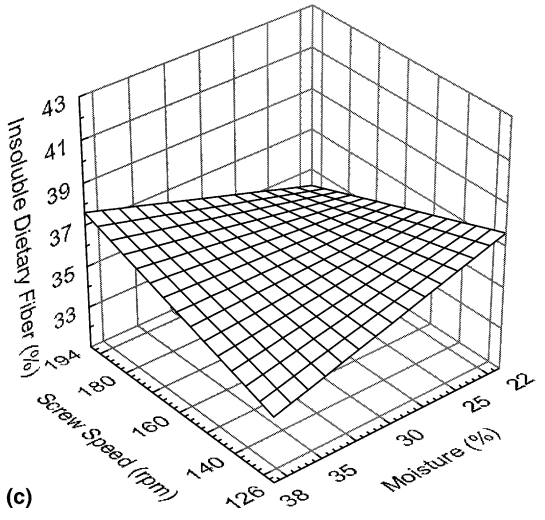


Fig. 1. (a) Effects of barrel temperature and moisture content on the content of insoluble dietary fibre of the orange pulp (screw speed 194 rpm). (b) The influence of barrel temperature and screw speed on the insoluble dietary fibre of the orange pulp (moisture content 30%). (c) The influence of moisture content and screw speed on the insoluble dietary fibre of the orange pulp (temperature 125 °C).

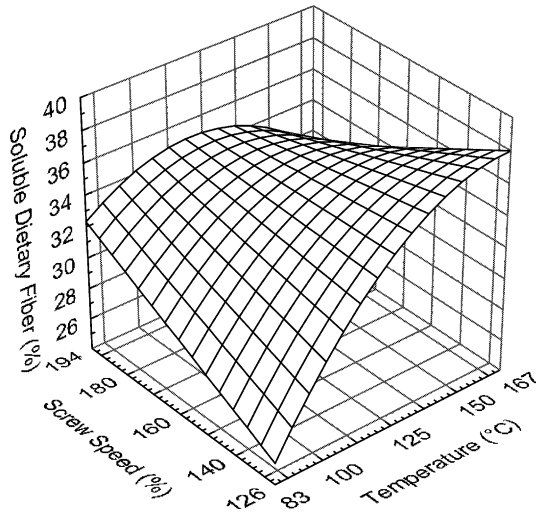


Fig. 2. The influence of barrel temperature and screw speed on the soluble dietary fibre of the orange pulp (moisture content 30%).

barrel temperatures were higher than 140 °C and screw speeds lower than 140 rpm, thus, fibre solubilization was favoured by longer residence times. This increment in SDF content would be caused by a partial solubilization of IDF, without completely degrading the polymeric structure (Ralet, Thibault, & Valle, 1991).The results found in this study agree with those previously reported (Anderson & Clydesdale, 1980; Roberston & Eastwood, 1981). Björck et al. (1984); Caprez, Arrigoni, Amado, and Neukom (1986); Ralet et al. (1991); Siljeström et al. (1986), who reported that, after extrusion processing, the values for SDF generally increased.

Gourgue et al. (1994) reported that the SDF contents of orange and lemon rinds increased after the extrusion process, from 10.19% to 22.46%, for orange, and from 28.09% to 37.67% for lemon.

Solubilization of the dietary fibre seems to depend on the conditions of the process and the method of analysis used. Thus, in processed wheat products with high feed speed and short residence time (high screw speed), solubilization was little evident (Björck et al., 1984). Varo, Laine, and Koivistoinem (1983) compared the values for FAS and found significant differences between the methods used.

3.5. Total dietary fibre

Fig. 3 shows that the TDF content decreased with higher barrel temperatures and lower moisture contents with the screw speed fixed at 160 rpm.

Ralet et al. (1991, 1993) reported that the TDF content of the extruded pulp of beetroot and pea rinds decreased by 7% and 5%, respectively, probably due to the fragmentation and solubilization of some polymers, which were not completely recovered during the precipitation with ethanol, thus sub-estimating the TDF content.

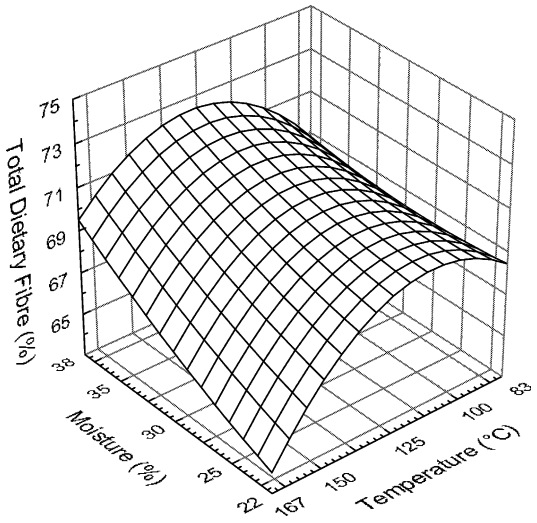


Fig. 3. The influence of barrel temperature and moisture content on the total dietary fibre of the orange pulp (screw speed 140 rpm).

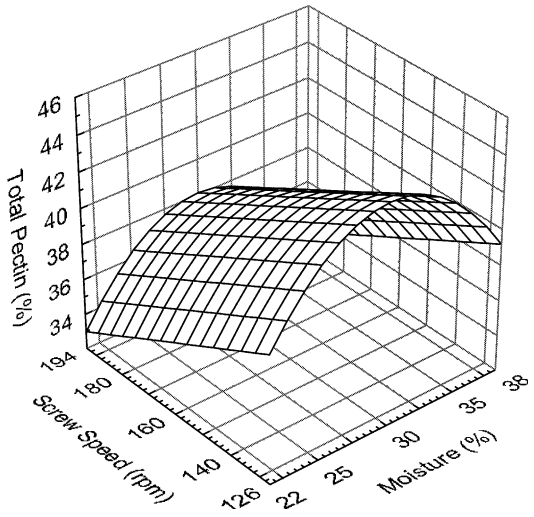


Fig. 4. The influence of moisture content and screw speed on the total pectin of the orange pulp (125 °C barrel temperature).

Also Siljeström et al. (1986) reported reductions in the TDF contents of extruded wheat flour, which were attributed to losses of arabinoxilan. Varo, Veijalainenk, and Koivistoinem (1984) reported that these components are sensible to thermal degradation under slightly acid conditions, very frequent during food processing.

Arrigoni, Caprez, Amado, and Neukom (1986) showed that the thermal processing of apple pulp decreased the TDF contents, possibly due to the solubilization and subsequent degradation of dietary fibre components, especially araban, favoured by the low pH. Thus, the thermal treatment under pressure of tomatoes at pH = 4.0, promoted reductions in the TDF, due to hydrolysis of fibre arabans.

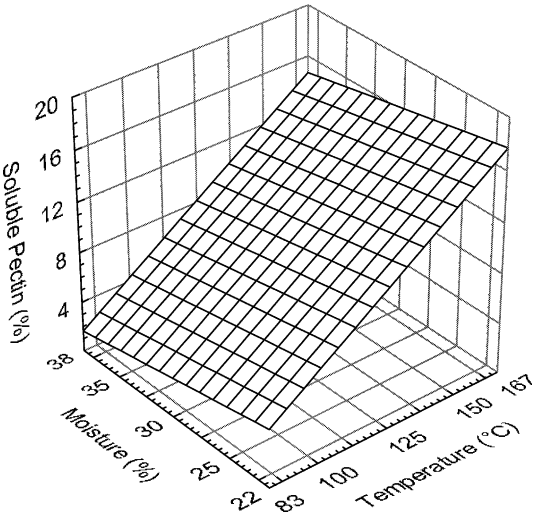


Fig. 5. The influence of moisture content and temperature on the soluble pectin of the orange pulp (125 °C barrel temperature).

4. Total and soluble pectin

Fig. 4 shows the effects of the moisture content (X_2) and the screw speed (X_3) for a fixed barrel temperature at the central point (125 °C). Higher values of TP can be obtained in the range of moisture content from 27% to 33% and lower values than 137 rpm of screw speed, or with longer residence times. Probably, these conditions are sufficient to cause alterations in the structure of the protopectin, allowing both solubilization and releasing the pectin (Ralet et al., 1991). Lower values for TP were evident under moderate conditions of processing, for shorter residence times (maximum screw speed) and higher moisture contents. However, if the severity of the process is increased by decreasing the moisture content (22%) and the screw speed remains maximum (194 rpm), the TP content also decreases. It is possible that the low moisture contents, associated with a higher degree of degradation caused by the greater mechanical shear resulted in some losses of the uronic acids, thus decreasing the TP content. All the extrusion assays promoted TP increases in the values. As previously mentioned, this fact could be attributed to a partial solubilization of the fibre components. This increment would also explain to a certain extent the increase observed in the SDF content. The fitted mathematical model for SP is shown in Fig. 5. Higher values for SP were reached under conditions of greater process severity caused by the highest barrel temperature (X_1) and the lowest moisture content (X_2) for a screw speed fixed at the central point ($X_3 = 160$ rpm). Contrary to these results, lower values of SP were found with lower barrel temperatures and higher moisture contents, when the processing conditions were less severe. Similar results were reported by Gourgue et al. (1994) who showed that extruded orange and lemon

rinds gave increased concentrations of acid sugars in the aqueous extracts, thus, confirming that the heat process solubilized pectic substances. Gourgue et al. (1994) reported that extrusion-cooking treatment increased the soluble fraction of dietary fibre as well as solubilised pectic substances and arabino-galactomannan chains. Also other staple fibre sources, such as beetroot pulp and pea rinds showed similar results (Guillon, Barry, & Thibault, 1992; Ralet et al., 1993).

Other researches showed that heat applied to a thin soup of broad beans, carrot and wheat bran, solubilized the pectic substances and, if their processing were severe, destruction could occur (Anderson & Clydesdale, 1980).

Ralet et al. (1991) observed that the insoluble residue in the water of beetroot pulp, after the extrusion process, showed relatively low contents of galacturonic acid and arabinose. However, values of 81% for galacturonic acid and 65.4% for arabinose were found in the aqueous extract, thus, indicating a marked solubilization of pectic substances during processing. Studies carried out by the same researchers (Ralet et al., 1993) reported that the uronic acids content in the aqueous extract of raw pea rinds (2.3%) increased drastically after twin-screw extrusion (21.5%). Simpson and Halliday (1941) studied the disintegration of the cellular wall of carrot during steam processing and reported that the soluble pectin content increased. On the other hand, the protopectin value showed a significant reduction, the reduction in the protopectin value being higher as compared to the increase in the pectin. These researchers indicated that if the processing conditions are severe, part of the pectin is destroyed.

5. Conclusions

The extrusion conditions (32.28–40.54%) decreased insoluble dietary fibre by 39.06% and increased soluble dietary fibre by 80%. Also, all the extrusion assays promoted total pectin values, attributed to a partial solubilization of the fibre components.

Higher values for soluble pectin were reached under conditions of greater process severity caused by the highest barrel temperature and the lowest moisture content. Lower values of soluble pectin were found with lower barrel temperatures and higher moisture contents, when the processing conditions were less severe.

The barrel temperature was the most important variable followed by the interactions between barrel temperature and screw speed, which affected the insoluble and soluble dietary fibre contents. Higher values for total (43.70%) and soluble pectin (16.76%) were reached with higher barrel temperatures and lower moisture contents due to solubilization of pectic substances.

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