Effect of Drying Temperatures on Starch-Related Functional and Thermal Properties of Acorn Flours

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Abstract: The application of starchy flours from different origins in food systems depends greatly on information about the chemical and functional properties of such food materials. Acorns are important forestry resources in the central and southern regions of Portugal. To preserve these fruits and to optimize their use, techniques like drying are needed. The effects of different drying temperatures on starch-related functional properties of acorn flours obtained from dried fruits of *Quercus rotundifolia* (QR) and *Quercus suber* (QS) were evaluated. Flours were characterized for amylose and resistant starch (RS) contents, swelling ability, and gelatinization properties. Drying temperature mainly affected amylose content and viscoamylographic properties. Amylograms of flours from fruits dried at 60 °C displayed higher consistency (2102 B.U. and 1560 B.U., respectively, for QR and QS). The transition temperatures and enthalpy were less affected by drying temperature, suggesting few modifications in starch structure during drying. QR flours presented different functional properties to those obtained from QS acorn flours. The effect of drying temperatures were more evident in QR.

Keywords: acorn flours, drying, functional properties, starch, thermal analysis

Introduction

Different plant sources (such as legumes, other seeds, tubers, and fruit pulps) can be regarded as an alternative raw material to flour production or as an ingredient to food products (Dendy and Dobraszczyk 2001). The use of flours from different sources in food systems depends greatly on the physicochemical and functional properties of such food materials. In spite of the impact of the functional properties on sensory quality and in consumer acceptability, limited information is available concerning the properties of acorn flour.

Acorn fruits from *Quercus suber* (QS) and *Quercus rotundifolia* (QR) are important forestry resources in the central and southern regions of Portugal. Traditionally, these fruits are mainly used for the feeding of Iberic pigs. In times of scarcity, the flours produced from these fruits were also used in bread production (Ribeiro 1992), and from these times some traditional recipes still remain. Other European Mediterranean countries are also consumers of these fruits, as stated by Rakic and others (2006).

Functional properties play important roles in the behavior of food or its ingredients during preparation, processing, and storage, affecting the sensory characteristics of foods. Kaur and Singh (2007) defined functionality as any property of a food ingredient, except its nutritional value, that has a great impact on its utilization. Those properties such as solubility, viscosity, water binding ability, emulsification, foaming, and gelation are of general interest (Hermansson 1979). The functionality of flours is closely related to the main chemical components which are determined by their genetic architecture, and fruits' postharvest conditions. Functional properties, such as forming, emulsification, nitrogen solubility, oil and water absorption are closely related to protein (Kinsella 1979), while viscosity and swelling characteristics are starch-related (Bressani 1985). However, Prinyawiwatkul and others (1997) emphasized that the gelling properties of heterogeneous systems, such as flour, are controlled both by physicochemical characteristics of proteins and starch components.

Starch-based materials have received considerable interest in recent years due to their biodegradability, renewability, and low cost (Russo and others 2007). Food producers pay particular attention to the major structural changes that occur as a result of heating during processing and lead to a change in the functional behavior of starch. In fact, the majority of starch applications involve the pasting of starch granules. Thus, proper understanding of the starch phase transitions or gelatinization is extremely important in food processing. As previously reported by (Correia and others 2009), for QR and QS, the same samples used in this present study, starch is the main component of acorn flours (49% for QR and 48% for QS), followed by sugars (26.4% for QR and 36.6 for QS), and fat (11.1% and 5.2% for QR and QS). It was also observed that fruit drying temperature affected the physicochemical properties of the fruit flours. However, functional properties were not assessed and we have no knowledge of any other studies.

Some well-established methods were used to determined important parameters, such as Brabender viscoamylograph test and the differential scanning calorimetry (DSC) analysis, to provide a deeper characterization of acorn flours functional and thermal properties some. Also, amylose content, resistant starch (RS) content, and swelling index were determined for further understand what change with drying fruit process, and in what way they contribute to starch paste characteristics.

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The present study aims to characterize starch-related functional properties of flours produced from acorn fruits dried at different drying temperatures, with a view to utilizing these flours in various food products.

Materials and Methods

Samples

QS and QR acorns were collected in "montados," located in Idanha-a-Nova (Centre East of Portugal). The acorns were harvested at full maturity. Total of 3 sets of 1 kg were randomly collected for each species. Samples were stored at 4 °C until experiments commenced. Fresh fruits (49% in moisture) were subjected to hand peeling to remove the tegument and pericarp. The nuts were chopped into little pieces, to further ease milling process. Pieces were then milled in a SK 100 Cross Beater Retsch hammer mill to pass through a 1-mm sieve. For fruits intended to be dried further, the drying process was conducted at different temperatures (40, 50, 60, and 70 °C) until a final a_w of 0.2 was reached, as described by Correia and others (2009). Then acorn fruits were milled in the same way as fresh fruits.

Chemical analysis

Moisture content was determined by gravimetric method at 100 to 105 °C, until a constant weight was reached.

The colorimetric method proposed by Juliano (1971), was used to determine amylose content, being amylose content expressed on starch basis. Total of 100 mg of each sample was weighed into a 50-mL Erlenmeyer flask and 1 mL of 95% ethanol and 9 mL of 1 N NaOH were added. The mixture was heated for 10 min in a boiling water bath to gelatinize the starch, cooled, and transferred, with several water washings, into a 100-mL volumetric flask. The volume was brought up with water and then mixed well. A total of 5 mL of starch solution was pipetted into a 100-mL volumetric flask and 1 mL of 1 N acetic acid and 2 mL of iodine solution (0.2 g iodine and 2 g potassium iodine in 100 mL of aqueous solution) were added. The solution is made up to volume with distilled water, shaken, and allowed to stand for 20 min. Absorbance of the solution at 620 nm was measured with a Lambda 25 UV/VIS spectrophotometer (PerkinElmer, Mass., U.S.A.). Amylose content was determined by reference to a potato amylose (Sigma Chemical Co., St. Louis, Mo., U.S.A.) standard curve.

RS content was determined following the method proposed by Mun and others (2006). Sample (1 g, db) was placed in a 50 mL screw cap centrifuge tube with a magnetic stirring bar and suspended in 20 mL sodium acetate buffer (pH 5.2). The sample tube was heated in a boiling water bath for 1 h with continuous stirring and immediately cooled to 40 °C. Enzyme solution (2 mL) was added and the mixture was incubated for 16 h at 37 °C in a water bath with stirring. After incubation, the reaction mixture was transferred to a 250-mL beaker with 117 mL of 95% ethanol, and allowed to stand for over 1 h at room temperature to precipitate the residue. The precipitate was collected in a tared sintered glass crucible (porosity nr 2). The insoluble residue was dried at 105 °C in an oven to determine the RS level. Enzymatic solution was prepared by the following method: 1 g of pancreatin (*Porcine pancreas*, Sigma Chemical Co.) and 12 mL of deionized water were placed in a centrifuge tube with magnetic stirring and mixed well. After centrifugation (1350 × *g*, 10 min), 10 mL of supernatant was mixed with 0.2 mL of pullulanase (*Promozyme*, Sigma Chemical Co.), and 1.8 mL of deionized water. RS level (%) was calculated as the ratio of the weight of insoluble residue to the initial sample weight.

All reagents used were of analytical grade. Results are expressed at a dry weight (db).

Swelling index

The swelling index of individual flours was determined with a method used by Mwangwela and others (2007). Samples of flours were dispersed in deionized water (1 : 20, w/ v) and stirred for 1 min, followed by heating in a water bath at 90 °C for 30 min with intermittent mixing. The heated samples were cooled for 30 s under running water and for 10 min in an ice-bath to accelerate gel formation. The tubes containing the gels were centrifuged (4500 × *g*, 20 °C) for 10 min. After this, samples rested for 5 min at 24 °C. The supernatant was decanted and the residue weighed. Swelling index was calculated as the ratio of the weight of the final residue to the initial sample weight.

Pasting properties

The gelatinization of acorn flours was monitored in a Brabender (Duisburg, Germany) viscoamylograph at 7% starch concentration, heating from 30 to 95 °C, holding at 95 °C for 15 min and then cooling until 50 °C. It should be noted that, for starch concentration lower than 7%, the paste consistency is too low and for higher concentration the paste consistency is extremely high giving viscoamylographic profiles with high variability.

Breakdown (BD), a measure of consistency breakdown extension, was calculated by Eq. 1 (Srichuwong and others 2005):

$$BD = ([\text{peak consistency} - \text{hot paste consistency at } 95\,C]/\text{peak consistency}) \times 100 \quad (1)$$

Scanning electron microscopy

The evaluation of starch granule modifications during the gelatinization process was evaluated by Scanning Electron Microscopy (SEM). Samples were taken at different heating stages (beginning of the peak, at maximum, and at the end) and sprayed onto double-sided tape on a microscope stub. Samples were analyzed by taking images on an environmental scanning electron microscope (ESEM) model Quanta 400 (FEI Co., Hillsboro, Oreg., U.S.A.), at 10 KV and 4 mbar.

Thermal properties

Thermal properties were evaluated by DSC on a Shimadzu calorimeter (model TA-50WSI, Kyoto, Japan). The instrument was calibrated using indium and purified, deionized distilled water as standards. The samples (10 mg) were weighed directly in DSC aluminum pans. Samples were added with water or allowed to equilibrate over a salt saturated solution to a final moisture content of 15% (a_w of 0.64 ± 0.035). The samples were submitted to heating over a range of temperatures from 25 to 180 °C at a heating rate of 10 °C/min, rate flow 30 mL/min, and nitrogen as carried gas. Onset temperature (T_o), peak temperature (T_p), end set temperature (T_e), and gelatinization enthalpy ΔH (J/g of dry starch) were determined. As the endotherms are essentially symmetrical, the total gelatinization temperature range (T_r), related with the heterogeneity of the starch granules crystallites, and peak height index (PHI), describe the relative shape of the endotherm, can be established by Eq. 2 and 3 (Krueger and others 1987):

$$T_r = (2[T_p - T_o]) \quad (2)$$

$$PHI = \Delta H/(T_p - T_o) \quad (3)$$

Statistical analysis

All the data are averages of at least 3 different determinations. Results were analyzed using the SPSS® for Windows version 18.0 and Statistic® version 6 software, whereby the data obtained were subjected to one-way analysis of variance (ANOVA) test. The separation of means or significant difference comparisons of all parameters were tested by Tukey's HSD test. Pearson correlation coefficients (r) for the relationships between properties were also calculated. Principal component analysis (PCA) and cluster analysis of the flours' properties were carried out to provide a ready mean of visualizing the differences and similarities among different samples. The level of significance used for all the statistical tests was 95%.

Results and Discussion

Amylose, RS, and swelling index

The effects of drying temperatures (DT) on starch-related functional properties are shown in Table 1. The drying temperatures positively affected amylose content ($r = 0.673$, $P < 0.05$), swelling index ($r = 0.746$, $P < 0.05$), and paste consistencies of the flours (at peak − $r = 0.773$, at 95 °C − $r = 0.769$, and at final − $r = 0.868$, $P < 0.01$). Furthermore, gelatinization temperature (GT) and temperature at maximum consistency are strictly correlated to DT. DSC does not seem to be an effective methodology to detect the effect of DT on functional properties, since no significant relationship was found. As shown in Table 2, the effect of drying temperature on amylose content is similar in both varieties, dried samples always presenting higher amylose content values. The observed increase of amylose content with drying temperatures may be due to the combined increased action of enzymes during the acorns' drying process in starch. Enzymes that show amylolitic properties are, mainly, α-amylase, β-amylase, glucoamylase, and pullulanase, which are active at the tested drying temperatures, with an optimum temperature lying between 55 and 60 °C (Matherwson 1998). This optimum may explain the highest amylose content values obtained with flours produced from fruits dried at 60 °C. Results showed that RS is closely correlated to amylose content ($r = 0.794$, $P < 0.01$, Table 1). Liu (2005) mentioned that the major commercial source of RS is that of high amylose starches; this compound being increased by heating processes. From the results show on Table 2 it is possible to observe that acorn flours presented high levels of RS, increasing with the drying temperature up to 60 °C. ANOVA showed that the RS content is only significantly different for flours of fruits dried at 60 °C.

Swelling index is important for certain products' characteristics, such as the moisture content of the product, starch retrogradation, and the subsequent of staling product (Siddiq and others 2010). The results showed that this parameter is significantly correlated with DT (Table 1). It was observed that swelling indexes are always quite high (Table 2) when compared with other starchy flours like cowpea (7.24%, Mwangwela and others 2007) and wheat (5%, Zaidul and others 2008). Higher swelling ability values are shown by flours dried upon 50 °C for QR and 40 °C for QS and the highest value observed for samples dried at 60 °C. This parameter

Table 1–Pearson correlation between chemical, functional, and thermal properties of acorn flours.[a]

	DT	AM	RS	SI	GT	PC	PT	C95 °C	CAH	FC	BD	To	Tp	Te	H	Tr
AM	0.673*															
RS	0.400	0.794**														
SI	0.746*	0.867**	0.620													
GT	−0.681*	−0.815**	−0.802**	−0.820**												
PC	0.773**	0.680*	0.159	0.650*	−0.362											
PT	−0.660*	−0.159	−0.094	−0.426	0.397	−0.576										
C95 °C	0.769**	0.691*	0.177	0.648*	−0.340	0.995**	−0.521									
CAH	0.732*	0.694*	0.203	0.565	−0.272	0.940**	−0.330	0.965**								
FC	0.868**	0.786**	0.583	0.804**	−0.689*	0.868**	−0.694*	0.872**	0.830**							
BD	0.478	0.429	0.087	0.497	−0.523	0.525	−0.692	0.449	0.265	0.435						
To	−0.061	0.238	0.545	0.109	−0.465	−0.267	−0.254	−0.304	−0.364	0.064	0.220					
Tp	−0.058	0.140	0.234	0.304	−0.142	−0.083	0.000	−0.052	−0.016	0.045	−0.348	0.243				
Te	−0.114	0.244	0.096	0.332	−0.217	0.005	−0.059	−0.016	−0.062	−0.030	0.241	0.395	0.710*			
H	0.368	0.195	−0.110	0.327	0.166	0.653*	−0.162	0.650*	0.643*	0.380	−0.168	−0.403	0.434	0.179		
Tr	−0.017	−0.008	−0.101	0.228	0.141	0.082	0.150	0.134	0.204	0.007	−0.465	−0.365	0.815**	0.445	0.659*	
PHI	0.280	0.071	−0.173	−0.101	0.137	0.384	−0.193	0.368	0.334	0.248	0.367	−0.114	−0.755*	−0.526	0.058	−0.655*

AM = Amylose; DT = Drying temperature; RS = Resistant starch; SI = Swelling index; GT = Gelatinization temperature; PC = Peak consistency; PT = Peak temperature; C95 °C = Consistency at 95 °C; CAH = Consistency after holding at 95 °C; FC = Final consistency; BD = Breakdown; T_o = Onset temperature; T_p = Peak temperature; T_e = Ending temperature; ΔH = Gelatinization enthalpy; T_r = Total gelatinization temperature range; PHI = Peak height index.
*$P > 0.05$, **$P > 0.01$.

is indicative of starch granule swelling during gelatinisation, as well as water retention due to protein gelation (Mwangwela and others 2007). Pomeranz (1985) mentioned that the major chemical compositions that enhance the swelling of flours are protein and carbohydrates since both constituents contain hydrophilic parts such as polar or charged side chains. Acorn flour, a matrix resulting from both the combination of different nutrients and the effect of drying temperatures on the chemical composition of these flours, was studied in a previous study (Correia and others 2009). Moreover, it was noticed that protein, lipid, ash, and fiber contents did not change for acorn flours obtained from fruits dried at different temperatures.

Pasting and thermal properties

Gelatinization is an order–disorder phase transition that involves diffusion of water into the starch granules, hydration and swelling, uptake of heat, loss of birefringence and crystallinity, and amylose leaching (Biliaderis and others 1980). However, the pasting pattern of a starch, in a heterogeneous system such as in flour, may be quite different from that obtained in a single-component system, like extracted starch (Henshaw and others 1996).

Acorn flours contain, as mentioned previously, other substances such as protein, lipid, sugars, and minerals, which that may interact with starch at different degrees. Along with amylose content, some noncarbohydrate constituents such as lipid and protein may change pasting characteristics (Mangala and others 1999). Protein and starch are known to interact during gelatinization due to the attraction of their opposite charges, forming complexes (Pomeranz 1985). Protein fractions may also influence the hydration rate of starch granules by binding water in competition with starch (Whistler and Daniel 1985). Eliasson and Krog (1985) stated that the presence of lipids retard firming and retrogradation of starch by changing the water activity of the system, or by inhibiting amylopectin crystallisation. Therefore, paste characteristics of acorn flours would be affected by these interactions and by other factors, such as the amylose and damaged starch contents.

As mentioned previously, drying temperature significantly affected the GT and paste consistencies. The pasting parameter values (Table 3) allowed the conclusion that all of the samples presented a stable apparent viscosity profile over time, similar to a viscoamylograph profile of crosslinked starch suspensions (Thomas and Atwell 1999). This behavior is more evident in fresh fruit flours and QS flour obtained from dried fruits at 70 °C, where a peak consistency (PC) is absent.

As already stated, GT is negatively correlated to DT, and it is considered as an index of crystallite perfection (Shi and Seib 1992). The effect of drying temperatures on the GT was not found to be significant (Table 3), meaning that drying temperatures did not induce a loss of crystallinity. With regard to PC it can be stated that the values generally increase with DT ($r = 0.773$, $P < 0.01$), probably due to a major preservation of the starch granules. This is corroborated by Thomas and Atwell (1999) who stated that the paste viscosity is highest when a majority of swollen, intact granules are presented in the paste. In contrast, the temperature at the maximum consistency was negatively correlated with DT ($r = -0.660$). A high-peak temperature indicates the presence of starch that is highly resistant to swelling. In our results, no significant differences were observed considering the drying temperatures of 40, 50, and 60 °C. Table 1 also showed, as expected, a high correlation between PC and the consistency at 95 °C, consistency after holding the temperature at 95 °C, final consistency, and swelling index (with correlation coefficients of 0.955, 0.940, 0.868, and 0.650, respectively).

BD is a measure of the ability of the swollen granules in the starch paste to resist thinning during prolonged heating and mechanical shear, giving an indication of paste stability (Fu and others 2008; Liu 2005). In the present study, BD was almost inexistent, showing stability in paste apparent viscosity. Flours from both fruits dried at 60 °C presented the lower BD values, 2.4% and 7.3%, respectively, for QR and QS (Table 3). The BD is more evident in QS flours.

Table 2–Effect of different drying temperatures on starch content, amylose content, and swelling index of acorn flours.[A]

Sample	Drying temperature (°C)	Amylose	Resistant starch	Swelling index
QR	None	41.7 ± 0.26e	35.5 ± 0.24d	9.1 ± 0.08b
	40	62.7 ± 0.35b	37.8 ± 0.75cd	9.3 ± 0.17b
	50	67.9 ± 0.15h	37.5 ± 0.87cd	10.2 ± 0.08c
	60	84.1 ± 0.49fg	39.9 ± 0.43bc	10.9 ± 0.10a
	70	56.5 ± 0.32c	36.0 ± 1.10d	10.3 ± 0.09c
QS	None	48.9 ± 0.46d	38.8 ± 0.37bcd	9.0 ± 0.06b
	40	71.5 ± 0.58a	38.8 ± 0.64bcd	10.7 ± 0.02ac
	50	82.5 ± 0.38g	41.6 ± 0.49ab	10.4 ± 0.02ac
	60	86.1 ± 0.12f	43.9 ± 0.58a	11.2 ± 0.27a
	70	66.8 ± 0.64h	40.8 ± 0.92abc	10.1 ± 0.13c

Values are expressed as mean ± standard error.
[A]Means sharing the same letters in a column are not significantly different from each other (Tukey's HSD test, $P < 0.05$).

Table 3–Pasting properties of flours produced from acorn fruits dried at different drying temperatures.[A]

Sample	Drying temperature (°C)	GT (°C)	PC (BU)	PT (°C)	C95 °C (BU)	CAH (BU)	FC (BU)	BD(%)
QR	None	70.0 ± 0.58c	*	*	505 ± 29.0b	570 ± 17.6c	950 ± 29.0c	*
	40	67.5 ± 1.32c	1120 ± 34.6e	87.7 ± 2.60ab	1047 ± 24.0e	1060 ± 34.5c	1673 ± 40.6e	6.5 ± 0.10c
	50	65.3 ± 1.20bc	1923 ± 26.0c	86.5 ± 1.14ab	1786 ± 46.7c	1655 ± 34.8e	2435 ± 35.0f	7.2 ± 1.23bc
	60	65.7 ± 1.20bc	2102 ± 31.8b	88.0 ± 1.70b	2053 ± 29.1a	2065 ± 37.6a	2667 ± 35.3d	2.4 ± 0.26a
	70	65.3 ± 0.33bc	2090 ± 52.0bc	79.7 ± 1.76a	1872 ± 40.6c	1553 ± 35.0ef	2472 ± 38.1df	10.2 ± 0.6*b*0
QS	None	69.0 ± 0.56c	*	*	690 ± 17.3f	762 ± 20.3h	1287 ± 52.1b	*
	40	62.3 ± 1.20ab	920 ± 23.3a	88.3 ± 1.59b	843 ± 26.0f	748 ± 26.0h	1506 ± 52.0e	8.4 ± 0.55bc
	50	60.3 ± 0.88a	1540 ± 25.0d	85.7 ± 2.46ab	1394 ± 28.5d	1377 ± 43.5g	2400 ± 31.8f	9.6 ± 0.57bc
	60	59.0 ± 1.15a	1560 ± 35.0d	82.5 ± 1.44ab	1447 ± 26.0d	1215 ± 29.0fg	2930 ± 49.3a	7.3 ± 0.38bc
	70	62.3 ± 0.67ab	*	*	1107 ± 58.1e	1244 ± 62.5b	2270 ± 49.0f	*

Values are expressed as mean ± standard error. Consistency is reported as BU (Brabender units). GT = gelatinization temperature; PC = Peak consistency; PT = peak temperature; C95 °C = consistency at 95 °C; CAH = consistency after holding at 95 °C; FC = final consistency; BD = breakdown.
[A]Means sharing the same letters in columns are not significantly different from each other (Tukey's HSD test, $P < 0.05$).
*No maximum peak.

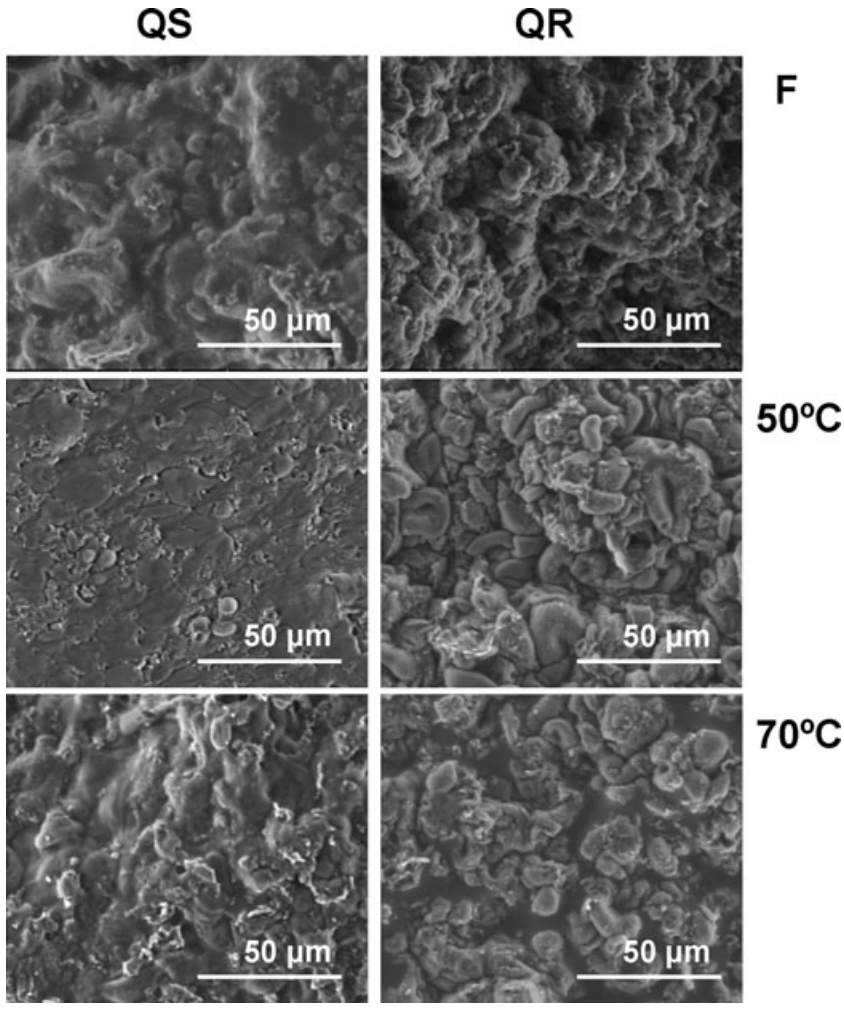

Figure 1–SEM photomicrographs of QS and QR samples collected at the beginning of gelatinization (F = Fresh; Drying temperatures = 50 and 70 °C).

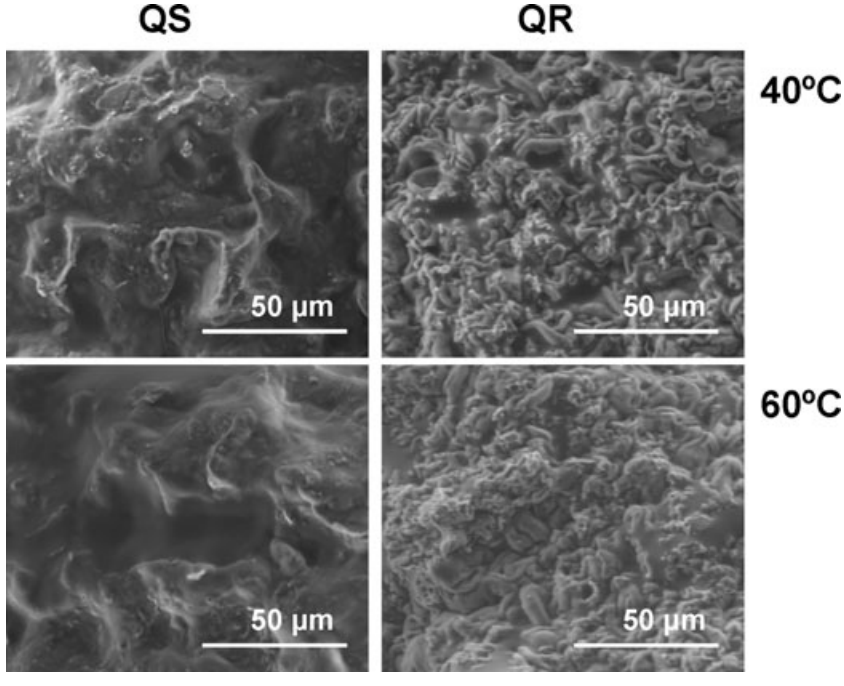

Figure 2–SEM photomicrograph of peak gelatinization for QS and QR dried flours, at 40 and 70 °C, during the amylographic tests (F = Fresh).

Table 4–Thermal (DSC) properties of dried acorn flours.

Sample	Drying temperature (°C)	T_o (°C)	T_p (°C)	$-\Delta H$ (J/g)	T_r (°C)	PHI (J/g °C)
QR	None	64.7 ± 1.91abc	119.8 ± 1.08c	17.7 ± 0.54c	110.2 ± 6.19bc	0.32 ± 0.02c
	40	64.4 ± 2.17abc	90.1 ± 1.25b	16.4 ± 0.24bcd	51.4 ± 2.26a	0.64 ± 0.06a
	50	54.4 ± 0.96c	93.7 ± 1.88ab	15.7 ± 0.34bde	78.7 ± 4.83d	0.52 ± 0.03ab
	60	55.7 ± 2.82c	117.6 ± 3.28cd	25.7 ± 0.31a	123.8 ± 3.78b	0.40 ± 0.01bc
	70	59.5 ± 2.51bc	103.9 ± 2.51ae	20.3 ± 0.66a	88.9 ± 1.94d	0.46 ± 0.01bc
QS	None	60.8 ± 3.13abc	102.0 ± 3.41abe	14.4 ± 0.16e	82.3 ± 4.37d	0.35 ± 0.02bc
	40	60.3 ± 2.40bc	106.6 ± 3.01de	14.1 ± 0.13e	92.5 ± 3.63cd	0.32 ± 0.02c
	50	71.2 ± 2.83ab	109.0 ± 3.89cde	14.9 ± 0.18be	75.6 ± 2.43d	0.37 ± 0.01bc
	60	72.4 ± 2.84a	112.7 ± 2.16cde	17.0 ± 0.24cd	80.6 ± 7.45d	0.42 ± 0.04bc
	70	61.1 ± 0.85abc	108.4 ± 1.22cde	16.9 ± 0.32cd	94.7 ± 3.63cd	0.36 ± 0.01bc

T_o = Onset temperature; T_p = Peak temperature; ΔH = Gelatinization enthalpy; T_r = Total gelatinization temperature range; PHI = Peak Height Index (Equation 3).
For each determined parameter values followed by the same uppercase letter are not significantly different at $P > 0.05$, Tukey's HSD test.

A positive correlation was found between DT and final consistency ($r = 0.868$, $P < 0.01$). At the end of the pasting process, the consistency of acorn pastes from acorn flours produced after drying at 60 °C was about twice that of pastes produced from fruits dried at 40 °C (Table 3). Again, this fact is probably related to a higher value of amylose content ($r = 0.786$; $P < 0.01$) and the reassociation between starch molecules, especially amylose, resulted in the formation of a strong gel on cooling to produce a paste that will set into a firm and cuttable gel.

Gel morphology during gelatinization and pasting was observed by SEM. The effect of drying temperature was more evident at the beginning and at the peak of gelatinization as shown by the representative images in Figure 1 and 2. The images of the other samples are not shown because they were very similar to the images already presented. When comparing SEM images of both species at the same gelatinization state it seems that QS flours melt faster than those of QR (Figure 1 and 2). The QR starch granules seemed to be more organized and resistant to heating temperature. QS flour pastes presented a uniform aspect, showing a complete starch granule gelatinization (Figure 2).

In spite of the fact that the DSC results did not correlate to DT, some effects were observed. In general, the T_o, T_p, and T_e of the gelatinization were always higher (Table 4) than those found by amylographic method. It should be kept in mind that thermal analysis detected transition phenomena, and so the interference of other compounds than starch such as protein and fiber could have significantly increased the competition for water, decreasing the amount of water available to the starch (Wang and Kim 1998).

High transition temperatures were observed (Table 4), indicating that more energy is required to start starch gelatinization (Sandhu and others 2007). The T_o was not significantly different for fresh and dried acorn flours. Furthermore, results showed that at ending temperature, T_e, values were always high, between 139.9 ± 1.7 °C and 159.6 ± 3.3 °C, the differences among species and drying temperatures being not significantly different ($P \geq 0.05$). Also for QS flours, the effect of drying temperature did not influence the T_p values and also the T_r and PHI values. The high range, T_r, for both acorns suggests the presence of crystallites of varying stability within the crystalline domains of the granule (Singh and others 2004). For QR, the T_p values for fresh fruit flour and flour from fruit dried at 60 °C are quite similar. This behavior was also observed for the enthalpy and T_r, suggesting that for this drying temperature the starch granules were less affected.

In our study all the endotherms were symmetrical, and the relative shape of the endotherm was determined, by the peak high index (PHI). For the same drying temperatures, QS always presented lower PHI values, which can be related to a lower structured starch matrix (Table 4). In spite of this, results revealed high ΔH

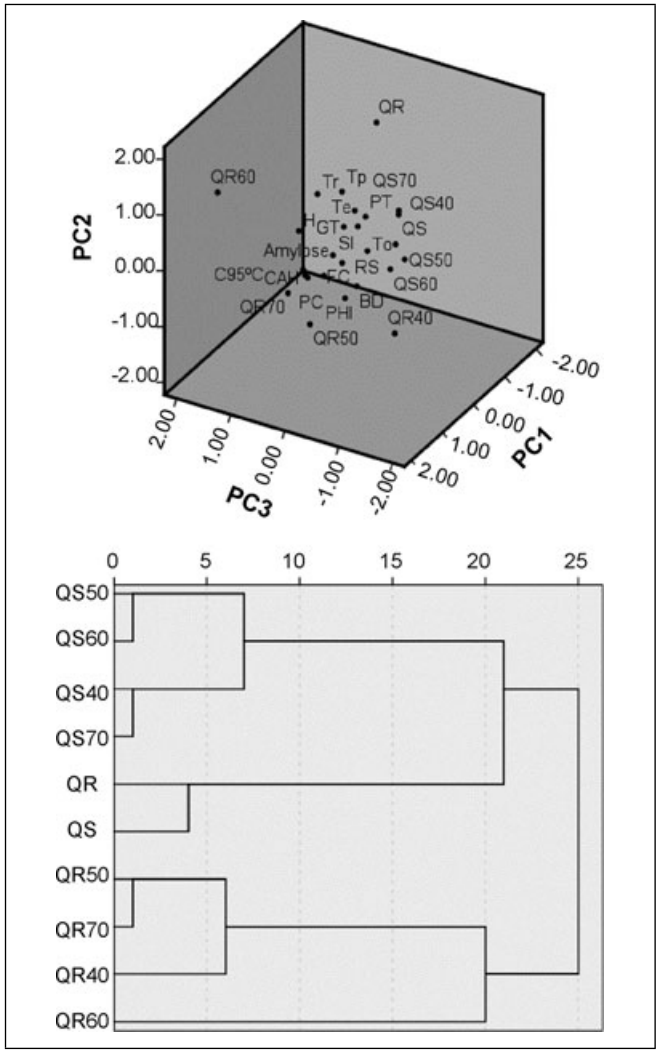

Figure 3–Principal component analysis (A): loading plot PC1, PC2, and PC3 for variables and samples. Cluster analysis (B) for flours considering the 4th components, the Ward method, and squared Euclidean distance as measure interval. QR = *Q. rotundifolia*; QS = *Q. suber*; numbers after these mentions are the temperature of drying process: 40, 50, 60, and 70 °C. RS = Resistant starch; SI = Swelling index; GT = Gelatinization temperature; PC = Peak consistency; PT = Peak temperature; C95 °C = Consistency at 95 °C; CAH = Consistency after holding at 95 °C; FC = Final consistency; BD = Breakdown; To = Onset temperature; Tp = Peak temperature; Te = end set temperature; H = Gelatinization enthalpy; Tr = Total gelatinization temperature range; PHI = Peak height index.

Table 5–PCA component loadings (unrotated).

	1	2	3
Amylose	0.895	−0.131	0.021
RS	0.831	0.208	−0.277
SI	0.522	0.341	−0.559
GT	0.877	0.325	−0.129
PC	−0.715	−0.237	0.596
PT	0.896	−0.212	0.329
C95 °C	−0.586	0.154	0.120
CAH	0.891	−0.183	0.379
FC	0.829	−0.139	0.460
BD	0.969	−0.039	−0.009
To	0.567	−0.324	−0.391
Tp	0.037	0.280	−0.810
C Te	0.065	0.955	0.166
ΔH	0.115	0.734	−0.107
Tr	0.422	0.243	0.773
PHI	0.042	0.748	0.644
Explained variation	7.183	3.454	3.101
Proportion of total variance	0.423	0.203	0.182

RS = Resistant starch; SI = Swelling index; GT = Gelatinization temperature; PC = Peak consistency; PT = Peak temperature; C95 °C = Consistency at 95 °C; CAH = Consistency after holding at 95 °C; FC = Final consistency; BD = Breakdown; T_o = Onset temperature; T_p = Peak temperature; T_e = end set temperature; ΔH = Gelatinization enthalpy; T_r = Total gelatinization temperature range; PHI = Peak height index.

values for flours, even for QS flours, which suggest that the double helices that unravel and melt during gelatinization are strongly associated with their native starch granules (Kaur and Singh 2007), and consequently more energy is required to melt their structure. The ΔH values were quite similar for acorn fresh fruit flours and acorn flours of dried fruits at 60 and 70 °C. When comparing enthalpy results of both acorns, it is quite apparent that for the same drying temperature, generally, QR starch granules need a higher level of energy to perform a transition (Table 4).

PCA was used to visualize the variation in functional, thermal, and pasting properties among the flours produced from acorn fruits dried at different temperatures. This statistical tool allows the reduction of a large number of variables to a few principal components (PCs) that describe the greatest variance in the analyzed data. All the studied properties results were subjected to PCA and the results are presented in Table 5 and Figure 3. Results showed that the space of 16 dimensions was reduced to a plane defined by 3 principal components, PC1, PC2, and PC3. Together, the PCs explain 80.8% (PC1—42.3%; PC2—20.3%; PC3—18.2%) of the total variability (Table 5). The loading factors showed that amylose and viscoamylographic properties were related to the PC1 and thermal properties were related to PC2 and PC3. Figure 3A shows a 3-dimensional projection of the evaluated properties and the flours of the 3 principal components (PC1, PC2, and PC3). The QR flours seem to be more related with the consistency properties in opposition of QS flours, as these are more related with thermal properties. From the analysis, it is possible to conclude that QR flour obtained from fruits dried at 60 °C presented quite different properties. Dendrograms produced by Cluster analysis show that at the same Euclidean distance (20) 2 subsets were found (Figure 3B). One includes dried QR samples (A) and the other includes the dried QS samples and both QS and QR fresh flours (B). In subset A, QR dried at 60 °C is clearly different from the other dried samples. In subset B, apart from fresh samples, the dried QS flours are clustered in the same group.

Conclusions

The aim of the present study was to assess the effect of drying temperature on functional properties of QS and QR acorns. Based on the results, it is possible to conclude that drying temperature affected starch-related functional properties, mainly the amylose content and the viscoamylographic properties. Drying fruits affected less QS flours starch-related functional properties than where it was applied to QR. However, improvement on functional properties was observed by drying QR fruits, mainly because of the higher paste consistencies and paste stability, leading to a resultant stable strong cutlet gel. This is more evident for flour from that fruits dried at 60 °C, suggesting that this flour is the flour that presents the best functional properties as a food ingredient.

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