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Raw and extruded fibre from pea hulls. Part I: Composition and physico-chemical properties

M.-C. Ralet, G. Della Valle & J.-F. Thibault*

Institut National de la Recherche Agronomique, Laboratoire de Biochimie et Technologie des Glucides, rue de la Géraudière, BP 527-44026 Nantes Cedex 03, France

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The effects of extrusion-cooking on pea hulls were investigated. Two batches of pea hulls of different average particle size (80 μm and 500 μm) were extruded under various conditions with a twin screw extruder (Clextral BC 45). In all cases, the water-solubility of the product increased (3·6–15·3%) with the severity of the treatment, monitored by the specific mechanical energy (115–363 kWh/t). A redistribution from insoluble dietary fibre to soluble dietary fibre, indicating a partial solubilisation of fibre without extensive degradation of the polymeric structure, was observed. The solubilised material was composed of pectic substances and hemicelluloses. The hydration properties of pea hulls differed much within the particle size distribution of the products and were not markedly modified by extrusion-cooking treatment.

INTRODUCTION

Numerous studies concerning physiological as well as technological aspects of dietary fibres have been carried out, and beneficial effects of these products are now well established. Up to now, wheat bran has been the major source of dietary fibre in human nutrition. However, other sources of fibre, especially some industrial by-products such as peanut hulls, sunflower hulls, pea hulls and sugar-beet fibres (Childs & Abajian, 1976; Sosulski & Cadden, 1982, Dreher & Padmanaban, 1983; Michel *et al.*, 1988) were investigated.

Pea hulls are particularly rich in dietary fibre, twice as much as wheat bran (Arrigoni *et al.*, 1986). They are lightly coloured and tasteless, which make them interesting sources of fibre. It has been shown that pea hulls were suitable to be incorporated in different kinds of bread (Satin *et al.*, 1978; Sosulski & Wu, 1988) and the nutritional interest of these products has been demonstrated (Longstaff & McNab, 1989; Hamberg *et al.*, 1989).

In previous studies, we have shown that extrusion-cooking of some dietary fibre-rich products (wheat bran, sugar-beet pulp, apple pomace and citrus peels) led to an important solubilisation of dietary fibre (Thibault *et al.*, 1988; Ralet *et al.*, 1990, 1991). The aim of this study was to investigate the effects of extrusion-cooking on physical properties and chemical composition of pea hulls.

EXPERIMENTAL

Material

Rough fibre (RF0) and ground fibre (GF0) of different average size, 500 μm and 80 μm, respectively, were obtained from Sofalia (Chappes, France).

Extrusion-cooking

A twin-screw extruder Clextral BC 45 was used. The 1 m long barrel included four heating and cooling zones. The screw configuration featured a positive displacement element with decreasing pitches and a reverse pitch element before the die head. The die was made of two cylindrical tubes (diameter 4 mm, length 30 mm). Electrical power of the screw motor drive was

*To whom correspondence should be addressed.

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measured in order to control the steady state of the extruder during sampling and to compute the specific mechanical energy (Della Valle *et al.*, 1989).

Operating conditions were varied by the screw speed (200–250 rpm) and the rate of added water (30–60% of solid feed rate). The feed rate was kept constant at 20 kg/h during the extrusion of RF0 whereas it was varied from 20 kg/h to 13 kg/h during the extrusion of GF0. Product temperature was measured before the die.

As extrusion-cooking treatment led to agglomeration of the fibre, the extruded samples were ground with a hammer mill (Culati) at a linear velocity of 100 m/s and passed through a 1 mm screen.

Extrusion conditions and nomenclature of the samples are shown in Table 1.

Aqueous extraction

Material (5 g) was stirred in distilled water (150 ml) for 30 min at 25°C. The suspension was centrifuged at 3000 g at room temperature for 15 min and the supernatant was collected. This extraction was carried out 3 times. The soluble fraction was filtered through G4 sintered glass, concentrated and freeze-dried. The residue was washed successively with ethanol and acetone, dried overnight at 40°C and weighed.

Analytical methods

Moisture content was determined by drying at 120°C for 2 h. All the compositions are given on a moisture-free basis. Protein content (N × 6·4) was determined by the Kjeldahl procedure. Ash was measured after incineration overnight at 550°C, then for 1 h at 900°C. Uronic acids (as galacturonic acids) were determined by the automated *m*-phenylphenol method (Thibault, 1979) in the soluble fractions and by the method of Ahmed & Labavitch (1977) in the insoluble fractions. Total neutral sugars were quantified by the automated orcinol method (Tollier & Robin, 1979). The individual sugars were reduced, acetylated and analysed by GLC (Blakeney *et al.*, 1983). Soluble fractions were hydrolysed by 2N trifluoroacetic acid (2 h, 121°C) while insoluble fractions were prehydrolysed by 72% sulphuric acid (1 h, 25°C) (Saeman *et al.*, 1954), diluted to 2N and heated (2 h, 100°C). Non-cellulosic glucose was measured on insoluble fractions after hydrolysis by 2N sulphuric acid (2 h, 100°C). Methanol and acetic acid were determined by HPLC on an Aminex HPX 87 H column (Voragen *et al.*, 1986). The differentiation between galacturonic and glucuronic acid was performed by HPLC (Aminex HPX 87 H) after acidic hydrolysis. Dietary fibre content was determined gravimetrically in duplicate after enzymic removal of starch and proteins (Prosky *et al.*, 1988).

Physico-chemical properties

The cation-exchange was determined by titration of 100 mg of sample with 0.02·N potassium hydroxide; the samples were previously converted into their acidic form by stirring 300 mg of product overnight at 4°C in 30 ml of 0·01N hydrochloric acid followed by extensive washing with distilled water until the conductivity of the washing was <5 μS.

Swelling was measured in duplicate by the bed volume technique (Kuniak & Marchessault, 1972). Dry fibres (100 mg) were weighed in a glass cylinder and left overnight at room temperature in distilled water. Results were expressed as millilitres of swollen sample per gram of dry initial sample.

The determination of water-holding capacity was carried out in duplicate on the Baumann apparatus (Baumann, 1967) with a 100 mg sample. Results were expressed as grams of water per gram of dry matter.

The water-binding capacity was determined in duplicate by centrifugation (MacConnell *et al.*, 1974). The samples were soaked overnight in distilled water (25 ml, 4°C) and centrifuged 1 h at 14 000 g. The

Table 1. Processing conditions and nomenclature of the pea hulls samples

	Temperature (°C)	Screw speed (rpm)	Water added (% of dry matter)	Feed rate (kg/h)	Temperature of the product (°C)	SME (kWh/t)
Raw pea hulls						
RF1	100	200	60	20	110	115
RF2	100	200	45	20	117	192
RF3	100	247	45	20	117	232
RF4	100	247	30	20	118	278
Ground pea hulls						
GF1	100	200	60	20	103	181
GF2	100	243	60	20	104	208
GF3	100	243	60	15	106	231
GF4	100	243	45	13	115	363

supernatants were carefully removed and the residues left for 1 h on G2 sintered glass. The samples were weighed, dried for 2 h at 120°C and weighed again. Results were expressed as grams of water per gram of dry residue.

RESULTS AND DISCUSSION

Particle size

As shown in Fig. 1, the particle size distribution of extruded RF0 after grinding was very different from that of the initial product. Compared to RF0, RF1 showed a large decrease of the 1000–500 μm and a large increase of the 500–250 μm population, while the two other populations (250–125 μm and <125 μm) were only slightly increased. Products extruded under more severe conditions (RF2, 3 and 4) had, compared to RF1, a decreased population between 500 μm and 250 μ and a simultaneous increase in the two finest ones. RF4 was particularly rich in particles under 125 μm. After grinding, the average particle size of extruded samples was always much lower than that of initial fibre and decreased when the severity of the extrusion treatment increased.

For GF, no significant change of average particle size (80–110 μ) was noted after extrusion-cooking.

Water solubility

The water solubility of RF0 was low (1·4%); it was slightly increased after grinding (GF0: 3·2%). After extrusion cooking of RF0, the water solubility markedly increased up to 15·3% for RF4, and was related to the specific mechanical energy (Fig. 2(a)). The water solubility of extruded GF increased from 7% to 14·8% with increasing specific mechanical energy. For specific mechanical energies between 150 kWh/t and 250 kWh/t, the increase in solubility observed for ground and rough pea hulls was similar. GF4 showed a water

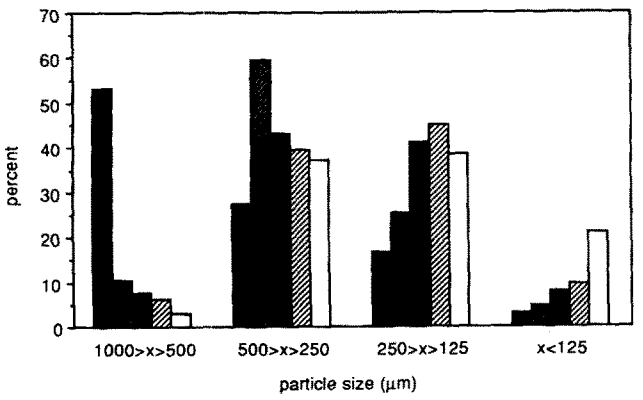

Fig. 1. Particle size distribution of rough pea hulls: ■, RF0; ▨, RF1; ▩, RF2; ▨, RF3; □, RF4.

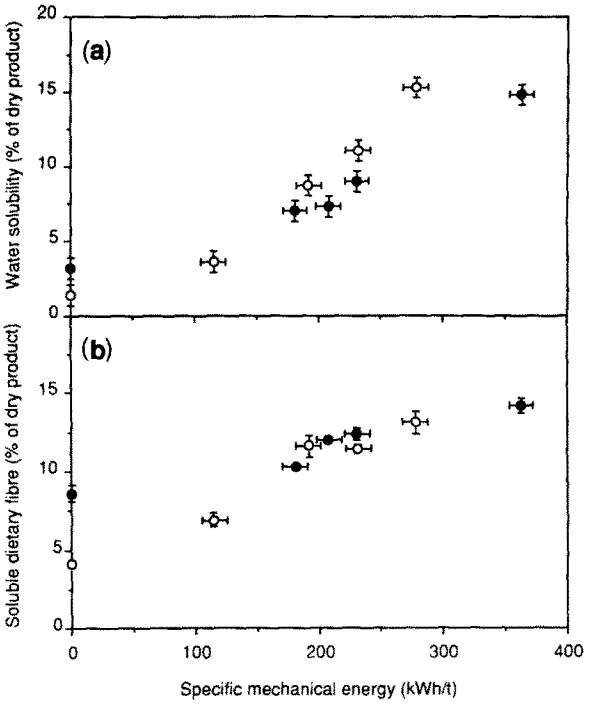

Fig. 2. (a) Pea hulls water solubility and (b) content in soluble dietary fibre as a function of specific mechanical energy: ○, rough pea hulls; ●, ground pea hulls.

solubility value of 14·8%, close to that found for RF4 (15·3%) in spite of the differences in specific mechanical energy (363 kWh/t and 278 kWh/t, respectively). This suggests the existence of a critical value (SME 280 kWh/t) of specific mechanical energy over which the solubility could not be increased whatever the energy supply. Another explanation might be that rough samples could be more sensitive to mechanical stress.

Chemical composition of the products

In agreement with previously published data (Longstaff & McNab, 1989), the pea hulls were mainly composed of glucose (45·1%; 98% of which was of cellulosic origin), xylose (14·6%) and uronic acids (12·7%; 99% of which was represented by galacturonic acid) (Table 2). The presence of galacturonic acid, rhamnose, galactose and methanol suggests the occurrence of an important pectic fraction. The composition of the fibres indicates that cellulose and xylose-containing polymers can be possibly the other polysaccharides. GF0, RF0 and their water-insoluble residues had close compositions. The fact that the water-insoluble residues of ground samples had lower contents in uronic acids than those from the rough samples suggested that the increase in water-solubility after grinding was due to the better solubilisation of pectic substances.

The soluble fractions were essentially composed of uronic acids, xylose and arabinose (Table 2). The relative content of uronic acids were always higher in

Table 2. Composition (% of dry matter) of pea hulls, insoluble and soluble fractions after water-extraction and extrusion-cooking

	Raw pea hulls										
	RF0	RF0		RF1		RF2		RF3		RF4	
		insoluble	soluble	insoluble	soluble	insoluble	soluble	insoluble	soluble	insoluble	soluble
Yield	100·0	98·6	1·4	96·4	3·6	91·3	8·7	89·0	11·0	84·7	15·3
Proteins	3·8	—	—	—	—	—	—	—	—	—	—
Rhamnose + Fucose	0·9	0·9	5·3	0·6	3·4	0·7	3·1	0·5	2·6	0·4	2·3
Arabinose	4·2	4·0	13·1	2·9	18·1	2·3	24·8	1·9	18·8	1·3	13·6
Xylose	14·6	14·9	19·8	11·8	31·3	13·0	26·2	11·6	21·6	10·0	21·3
Mannose	1·0	1·1	0·0	0·8	0·0	0·8	0·0	0·8	0·0	0·6	0·0
Galactose	1·2	0·9	7·7	0·8	3·5	0·7	4·8	0·6	3·6	0·4	3·6
Glucose	45·1	43·2	7·0	46·2	5·6	45·3	2·6	46·0	2·2	48·7	3·7
Uronic acids	12·7	13·0	19·8	12·8	28·4	13·9	22·7	11·4	21·0	12·1	18·2
Ash	1·7	—	—	—	—	—	—	—	—	—	—
Methanol	0·5	—	4·3	—	3·5	—	3·6	—	3·0	—	2·5
Acetic acid	1·0	—	3·1	—	3·6	—	3·6	—	2·2	—	3·4
	Ground pea hulls										
	GF0	GF0		GF1		GF2		GF3		GF4	
		insoluble	soluble	insoluble	soluble	insoluble	soluble	insoluble	soluble	insoluble	soluble
Yield	100·0	96·8	3·2	93·0	7·0	92·7	7·3	91·0	9·0	85·2	14·8
Rhamnose + Fucose	0·6	0·7	2·0	0·6	2·1	0·7	2·6	0·3	2·7	0·4	3·1
Arabinose	3·1	3·4	10·3	2·4	14·9	2·2	17·3	2·2	20·1	1·4	17·6
Xylose	11·2	12·3	17·9	12·1	19·9	10·9	23·9	10·5	24·2	10·3	21·4
Mannose	0·2	0·0	0·0	0·2	0·0	0·2	0·0	0·2	0·0	0·2	0·0
Galactose	1·0	0·9	4·2	0·8	3·8	0·6	4·8	0·8	4·7	0·5	4·5
Glucose	47·6	48·8	1·7	51·8	1·6	46·4	1·6	47·7	2·0	52·2	3·4
Uronic acids	13·9	10·2	32·6	9·4	31·8	9·3	31·5	9·4	31·4	7·7	25·2

the soluble fractions of GF than in RF. Eleven percent of the deoxysugars, 4·9% of the arabinose, 2% of the xylose, 11% of the galactose, 0·9% of the glucose and 2·3% of the uronic acids were recovered in the soluble fraction of RF0. These values remained unchanged in GF0 for rhamnose, galactose and glucose but were markedly increased for uronic acids (7·5%), for arabinose (10·6%) and for xylose (5·1%). The proportions recovered in the soluble fractions increased, from RF0 to RF4, from 11% to 57·1% for deoxysugars, 4·9% to 65·6% for arabinose, 2% to 28% for xylose, 11% to 55·6% for galactose and from 2·3% to 21·5% for uronic acids, and remained constant (0·9%) for glucose. The same features could be observed for GF. Except for xylose, which showed a plateau at about 280 kWh/t, the percent of each sugar recovered in the soluble fractions was linearly related to the specific mechanical energy (r^2 = 0·93) (Fig. 3). The slopes for solubilisation of rhamnose, galactose and arabinose were roughly similar (0·26, 0·23 and 0·26, respectively) whereas they had much lower values for xylose and uronic acids (0·1 and 0·075, respectively), and close to 0 for glucose. These results indicate that extrusion-cooking leads to the solubilisation of neutral pectic substances or pectic side-chains, rather than that of rhamnogalacturonans or hemicellulosic polymers, and that the cellulosic backbone was not degraded. Moreover, the better solubilisation of rhamnose compared to galacturonic acid suggests that rhamnogalacturonic 'hairy' regions are solubilised preferentially to 'smooth' homogalacturonic regions.

Dietary fibre content

The dietary fibre content of raw and extruded samples are given in Table 3. GF0 and RF0 showed a very high content in dietary fibre (91·5%), value in agreement with those of Caprez *et al.* (1987) and of Arrigoni *et al.* (1986). A decrease in the total dietary fibre content by 2% to 5% was found for the extruded RF, while this reduction reached only 1·7% for the extruded GF. It is likely that the severe extrusion conditions of RF4 and GF4 solubilised and fragmented some polymers which could not be fully recovered by the ethanolic precipitation during the analysis. Grinding the initial rough product led to an increase (110%) in the soluble dietary fibre. The extrusion-cooking treatment increased the soluble dietary fibre fraction by a partial solubilisation

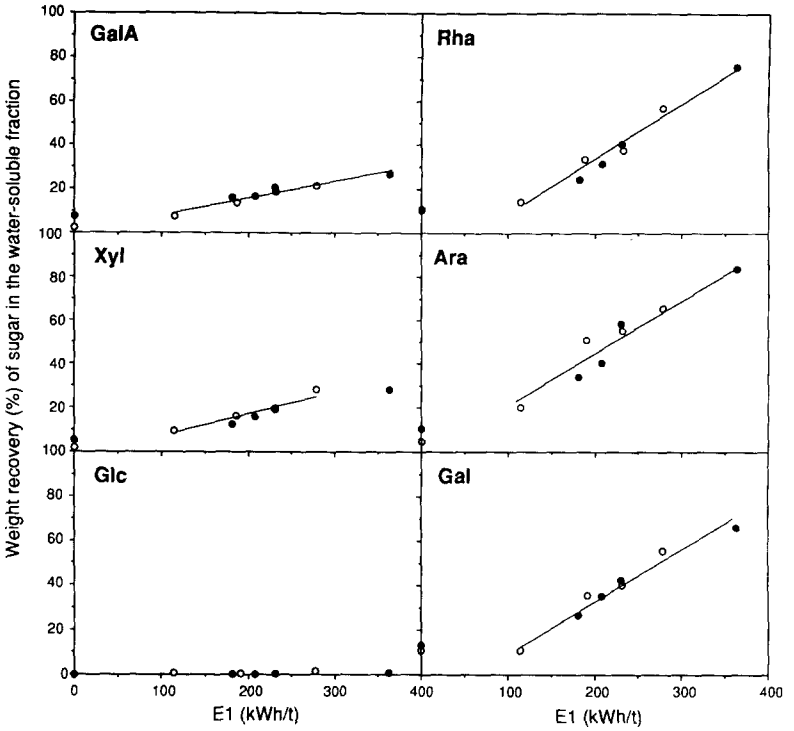

Fig. 3. Solubilisation of neutral and acidic sugars as a function of specific mechanical energy: ○, rough pea hulls; ●, ground pea hulls.

Table 3. Dietary fibre (DF) content of raw and extruded pea hulls (% of dry matter)

	Insoluble DF		Soluble DF		Total DF	
	Mean	SD[a]	Mean	SD	Mean	SD
Raw pea hulls						
RF0	87·4	(0·5)	4·1	(0·1)	91·5	(0·5)
RF1	82·6	(0·2)	6·9	(0·4)	89·5	(0·2)
RF2	77·7	(0·2)	11·6	(0·7)	89·3	(0·9)
RF3	77·0	(0·1)	11·4	(0·3)	88·4	(0·2)
RF4	73·4	(0·1)	13·1	(0·7)	86·5	(0·7)
Ground pea hulls						
GF0	83·0	(0·8)	8·6	(0·5)	91·6	(0·3)
GF1	80·9	(0·2)	10·3	(0·2)	91·2	(0·3)
GF2	79·5	(0·2)	12·0	(0·2)	91·1	(0·4)
GF3	78·2	(0·2)	12·4	(0·4)	90·6	(0·2)
GF4	75·7	(0·4)	14·2	(0·5)	89·9	(0·1)

[a]Standard deviations (n = 2)

of fibre without extensive degradation of the polymeric structure. The proportion of soluble fibre increased by 68% to 220% for RF and by 20% to 65% for GF. As shown in Figure 2(b), the soluble dietary fibre content of extruded samples increased with the specific mechanical energy and reached a plateau at nearly 15% of soluble dietary fibre.

The higher content in soluble dietary fibre than in water-soluble fraction at 25°C observed for the initial products and for those extruded under mild conditions clearly indicated that the method used for the dietary fibre analysis led to a degradation and a solubilisation of cell-wall polymers. The use of high temperature for both amylolysis and proteolysis (Prosky *et al.*, 1988) may explain this overestimation of the soluble dietary fibre content.

Physico-chemical properties

The samples behaved as monofunctional cation-exchangers. The cation exchange capacities were in the same range for all the samples (0·54–0·58 meq OH^-/g)

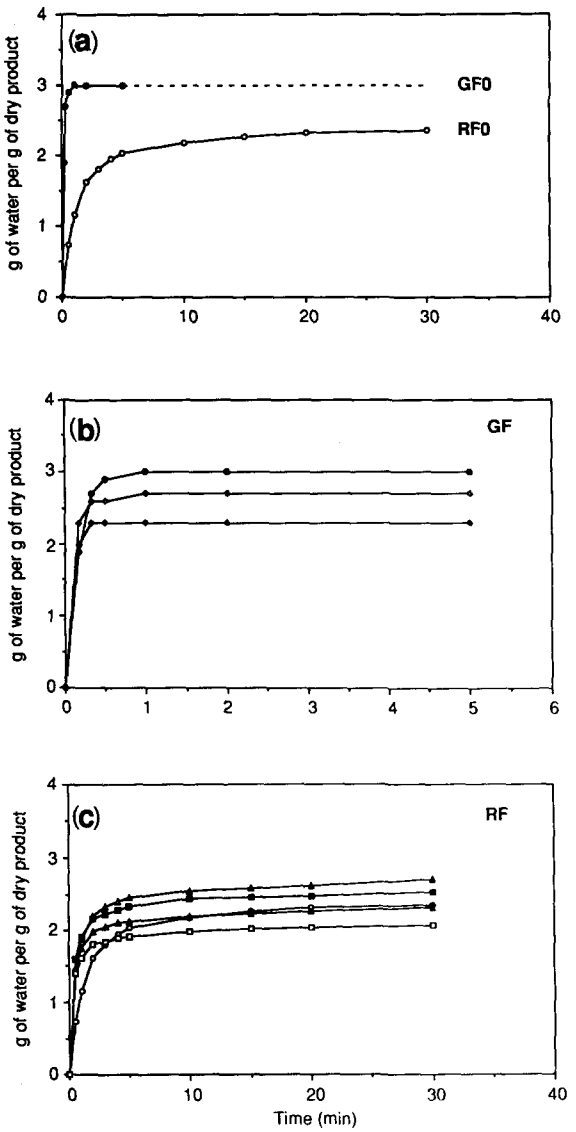

Fig. 4. Water-holding capacity (ml/g) of initial and extruded pea hull samples: (a) ○, RF0; ●, GF0; (b) ●, GF0; △, GF1; ▲, GF2, GF3, GF4; (c) ○, RF0; △, RF1; ■, RF2; ▲, RF3; ○, RF4.

in agreement (RF0 = 0·58 meq/g) with the value calculated from the amount of free carboxyl groups. The losses in uronic acids during the preparation of the acidic forms for RF2, RF0, GF3 and GF0 were only 2·3%, 0·3%, 2·4% and 0·9%, respectively.

The swelling values (Table 4) of RF0 and GF0 were rather low (6 ml/g and 4·7 ml/g) compared to those of other types of vegetable fibres (Rasper, 1979). Extruded RF0 showed lower values than RF0 when the extrusion-cooking treatment is severe. The swelling of GF0 was slightly lower than that of RF0 and was not modified by extrusion-cooking; the values were similar to those found for RF2, RF3 and RF4. The decreased swelling of extruded RF might be related to their lower average particle size.

RF0 and RF1 showed a water binding capacity of approximately 7 ml/g (Table 4). After the extrusion treatments of the highest severity (RF2, 3 and 4), the water binding capacity was diminished (4·3 ml/g, 3·9 ml/g and 3·5 ml/g respectively). GF0 showed a water binding capacity of 4·6 ml/g, which was not significantly modified after extrusion-cooking. This value was close to that (4·3 ml/g) of Sosulski & Wu (1988). Decreasing the particle size led to decreased swelling and water binding capacity probably due to the collapse of the fibre matrix.

The kinetics of water holding capacities of initial and extruded pea hulls measured with the Baumann apparatus are shown in Fig. 4. RF0 had a water holding capacity of 2·4 ml/g. GF0 showed, after 2 min, a water holding capacity of 3 ml/g with a very high initial rate of water uptake. The water holding capacity was slightly increased for RF extruded under mild conditions (RF1 and RF2) and slightly decreased for samples processed under more severe conditions (RF3 and RF4). The initial rate of water holding capacity was higher for the extruded samples. The same observations were made for extruded wheat bran (Ralet *et al.*, 1990). After extrusion-cooking of GF, the water holding

Table 4. Physico-chemical properties of initial and extruded pea hulls

	Cation exchange capacity (meq/g)	Water binding capacity (ml/g)		Water holding capacity (ml/g)		Swelling (ml/g)	
Raw pea hulls							
RF0	0·58	7·1	(0·1)[a]	2·4	(0·1)	6·0	(0·2)
RF1	—	7·3	(0·2)	2·7	(0·2)	5·6	(0·2)
RF2	0·55	4·3	(0·3)	2·5	(0·1)	5·2	(0·1)
RF3	—	3·9	(0·1)	2·3	(0·1)	4·9	(0·3)
RF4	—	3·5	(0·4)	2·1	(0·2)	5·0	(0·1)
Ground pea hulls							
GF0	0·54	4·6	(0·2)	3·0	(0·2)	4·7	(0·3)
GF1	—	4·5	(0·1)	2·7	(0·1)	5·1	(0·1)
GF2	—	4·5	(0·3)	2·3	(0·2)	5·2	(0·2)
GF3	0·54	3·9	(0·1)	2·3	(0·1)	4·8	(0·2)
GF4	—	4·3	(0·3)	2·3	(0·2)	4·8	(0·1)

[a]Values in parenthesis are standard deviations (n = 2)

capacity slightly decreased with increased specific mechanical energy. It is likely that grinding the products led to increased water holding capacities by increasing the surface area. On the other hand, for similar average particle size values, extruded samples showed decreasing water uptake with increasing severity of the treatment. For RF1 and RF2, the effects due to a mild extrusion-cooking treatment might be balanced by their lower average particle size. For RF3 and RF4, the severe extrusion-cooking treatment might induce a moderate collapse of the structure.

CONCLUSIONS

Physical properties of extruded pea hulls, and particularly their hydration properties, are not markedly modified by extrusion-cooking. Water-binding capacities of wheat bran were also not significantly modified by extrusion-cooking (Ralet *et al.*, 1990) while swelling and water-binding capacities of sugar-beet pulps were decreased after treatments of high severity (Ralet *et al.*, 1991). Arrigoni *et al.* (1986) have shown that water-binding capacities of pea hulls were only slightly modified after autoclaving while water-binding capacities of apple pomace were significantly decreased by this treatment. An explanation of these different behaviours could be that by-products consisting of primary cell walls are more fragile and that collapses due to the solubilisation of cell wall polymers could appear after treatments such as extrusion-cooking or autoclaving. By-products rich in secondarized cell walls, such as pea hulls or wheat bran, have a higher mechanical resistance and could be less sensitive towards collapses occurring during heat treatments.

The major effect of extrusion-cooking on pea hulls is a significant transformation of insoluble dietary fibre in soluble dietary fibre. However, the solubilisation of cell-wall polymers after extrusion-cooking is quite moderate for pea hulls compared to sugar-beet pulps (Ralet *et al.*, 1991). The structure of the cell walls seems to play a major role in the solubility of extruded by-products as pectic substances present in the primary cell walls of sugar-beet pulps are solubilised to a much larger extent than those present in highly cellulosic pea-hull cell-walls. The increase in the water-solubility of each sugar constitutive of pea hulls is well related to the specific mechanical energy. Extrusion-cooking seems to induce a preferential solubilisation of pectic 'hairy' regions compared to homogalacturonic pectic regions and hemicellulosic polymers.

The solubilised material is rich in pectins. Because of the well known nutritional effects (Jenkins *et al.*, 1976, 1978; Ebihara & Kiryiama, 1982; Vachon *et al.*, 1988) and potential functionality of these polymers, the structure of the solubilised material has to be studied. This will be described in the next paper (Ralet *et al.*, 1992).

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