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Effect of high pressure on the structure of potato starch

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Abstract

Potato starch–water suspension (10%) was subjected to high pressure treatment at 600 MPa for 2 and 3 min. Cross-polarization/magic angle spinning (CP/ MAS) ^{13}C NMR spectra of the obtained starch preparations indicated distinct signal resonances at 81.23 and 82. 46 ppm, respectively, which corresponded to amorphous sites (C-4) within starch structure. The F.t-i.r. analysis of starch preparations showed that high pressure significantly affected the intensity of bands corresponding to the amorphous (1018 cm^{-1}) and more ordered (1047 cm^{-1}) part of starch structure. The DSC analysis showed a decrease in gelatinization temperatures (T_o, T_p, T_c) upon high pressure treatment. A substantial decrease in the total enthalpy of pressurised starches along with the time of treatment was also found. The SEM analysis indicated that in the starch structure the granule's surface was the most resistant to high pressure treatment. The inner part of the granule was almost completely filled with gel-like network, with empty spaces growing in diameter towards the centre of the granule.

Keywords: Potato starch; High pressure; NMR; F.t-i.r.; Structure

1. Introduction

Starch is a naturally occurring biopolymer in which glucose is polymerised into amylose and amylopectin forming densely packed, semicrystalline structure of granule. Starches from different botanical sources contain from 15 to 30% of amylose, although there are some mutant wheat varieties which can produce from 1.2 to 39.5% of amylose (Bocharnikova et al., 2003) or corn varieties with 100% of amylopectin ('waxy' starch) and up to 75% of amylose ('amylose extender') (Gidley & Bociek, 1985). It is well known that starches of different botanical origins vary also in their size and shape as well as physico-chemical properties. The most important factor differentiating the physico-chemical properties of starches is their chemical and physical structure, i.e. degree of polymerisation, amylose to amylopectin ratio as well as diffraction pattern and the differences in crystalline region of granules (thickness and number of crystalline lammellae) (Błaszczak et al., 2003; Yuryev et al., 2004). Recently, many studies have focused on the structure of starch granule in respect of a better understanding the mechanism of starch gelatinisation, retrogradation and stability of starch gels as well (Błaszczak, Fornal, & Lewandowicz, 2001; Rubens & Heremans, 2000). The gelatinisation temperature is reached when the crystalline regions in starch granules start to destabilise, which leads to an irreversible loss of the granule structure. The gelatinisation patterns of starches depend not only on their chemical structure but also on the level and kind of energy supplied. Douzals, Marechal, Coquille, and Gervais (1996) reported that the phase transition of starch can be observed when the starch–water suspension is heated at 60–70 °C. Fornal, Błaszczak, and Lewandowicz (1998) reported that the starches with high amylose content require more energy during the course of gelatinisation.

Stolt, Stoforos, Taoukis, and Autio (1999) reported the high pressure to evoke starch gelatinisation already at room temperature. Douzals et al. (1996) found that wheat starch gelatinisation began at the pressure over 300 MPa, and at the level of 600 MPa complete gelatinisation was induced. It was also demonstrated that the B-type starches were more

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pressureresistant (Stute, Klingler, Bogusławski, Eshtiaghi, & Knorr, 1996), i.e. potato starch needed the pressure of 800–1000 MPa to reach total gelatinization (Kudła & Tomasik, 1992). These authors concluded that the A-type starches are the most sensitive and the C-type ones are located in-between B- and A-starches. The high pressure caused reversible hydration of the amorphous phase followed by irreversible distortion of the crystalline region, which in turn led to the destruction of the granular structure. It was shown by Stute et al. (1996) and Rubens and Heremans (2000) that the effect of high pressure on the mechanism of starch gelatinisation and granule structure is not alike as compared to the heat-water treatment. The manner of starch polymer chains packing in the granule structure is regarded decisive for starch behaviour under high pressure (Rubens & Heremans, 2000). These authors claimed also that the degree of the crystalline structure melting at high pressure might have depended on the type of starch (amylose–amylopectin ratio) being responsible for specific interactions which determine starch behaviour.

Stolt, Oinonen, and Autio (2001) found that under pressure barley starch formed pastes with creamy texture similarly to wheat, corn, tapioca and pea starches. They also reported that the properties of starch pastes and gels obtained under high pressure differed from those of the heat-gelatinised ones. It could have been linked to the restricted swelling of starch granules under high pressure conditions (Stute et al., 1996). Stolt et al. (2001) explained that phenomenon, pointing out the role of the stabilising effect of amylose under high pressure. Waxy maize starch disintegrated under high pressure, whereas amylomaize starch did not change its granular structure even at 900 MPa. High pressure was also found to affect rheological properties of barley starch, however starch granules remained intact and no leaching of amylose was found (Stolt et al., 2001).

Only few studies into the effects of high pressure on the changes in the potato starch structure have been undertaken so far. They were mainly focused on the effect of high pressure on potato starch dispersion. Stute et al. (1996) reported that 900 MPa at 50% moisture evoked an incomplete gelatinization of potato starch. They found that pressure treatment (600 MPa/15 min) of potato starch (5% suspension) did not affect the granule structure. Potato starch granules retained birefringence under polarised light and B-type crystallinity pattern. Douzals et al. (1996) found that a calorimetric analysis of pressure-treated (100–600 MPa, 50 MPa s^{-1}) water-starch suspension (16%, d.m.b.) gave no relevant modification of the gelatinization enthalpy of potato starch.

It is generally believed that high pressure technology offers a new possibility of starch application in food products, for example as fat substitute in low-energy food. For this reason, major efforts have been directed towards studying the effect of high pressure on the structure–physico-chemical properties relationship of starch modified this way (Douzals, Perrier-Cornet, Gervais, & Coquille, 1998; Rubens & Heremans, 2000; Stute et al., 1996; Stolt et al., 2001).

The aim of this study was to investigate the changes in the structure of potato starch ‘preparation’ obtained after potato starch suspension treatment, in excess of water, under high (hydrostatic) pressure as a function of time.

2. Material and methods

2.1. Material

The experimental material was the commercial potato starch (Superior Standard) obtained from the Starch and Potato Products Research Laboratory in Luboń, Poland.

2.2. Sample preparation

The pressure treatment was performed in excess of water, i.e. using 10% (w/w) starch–water suspension. The suspension was closed into teflon tubs (10 mL), precisely mixed, deaerated, closely sealed and pressure-treated.

2.3. Pressure treatment

The pressure treatment of starch–water suspensions was performed in a high pressure device (LV30/16, The Centre of High Pressure Analysis, Polish Academy of Sciences, Warsaw, Poland). The teflon tubes were put into a high pressure chamber (with the capacity of approximately 25 cm^3) filled with pressure transmitting medium which also minimised adiabatic heating. The samples were pressure-treated at the level of 600 MPa for 2 and 3 min at a rate of approximately 10 MPa/s^{-1}. The temperature inside the pressure chamber averaged 20 ± 2 °C. The pressure treatment was performed in two repetitions for each combination.

After the pressure treatment, the starch pastes and gels were frozen in liquid nitrogen and freeze-dried. The obtained starch products were used for F.t-i.r., NMR, DSC and SEM analyses.

2.4. F.t.-i.r spectroscopy

All the F.t-i.r. spectra were obtained on a Burker IFS 113 v spectrometer. The measurements were performed using a KBr tablet (Merck, <50 μm). The spectra were obtained in the range of 4000–450 cm^{-1}. Prior to recording, the spectra were transformed against a KBr background.

2.5. C CP/MAS NMR spectroscopy

The NMR experiments were performed in a Bruker CXP-300 spectrometer (Bruker Analytik GmbH, Karlsruhe, Germany) with a standard Bruker probe head. Solid-state cross-polarization/magic angle spinning (CP/MAS) carbon-13 spectra were obtained at 75.46 MHz. Spinning rates of 3–3.5 kHz, spin locking and decoupling fields of 50 kHz were used. Spectra were referenced to external Me_4Si via the residual 'rotor' peak (from perdeuterated PMMA) at 44 ppm. A contact time of 1 ms was applied to all the standard spectra with a recycle time of 2 s. At least 1000 scans were averaged for each spectrum. Other parameters were as follows: spectral width 30 kHz; acquisition time 30 ms; time domain points 2 K; transformation size 8 K, line broadening 20 Hz.

2.6. DSC analysis

The thermal transition of native and pressure-treated starches was investigated with the use of Mettler TA4000 DSC, operated under nitrogen. The samples were weighed in an aluminium pan and water was added to obtain a starch/water ratio of approximately 1:3. The pan was sealed, equilibrated for 30 min and heated at temperatures from 30 to 95 °C at the rate of 10 K min^{-1}. Characteristic temperatures of the transition were onset geletinisation temperature—T_o, peak temperature—T_p. The enthalpy of gelatinization—ΔH was calculated into the dry mass of the sample.

2.7. SEM analysis

The microstructure of starch gels and pastes after pressure treatment was examined with scanning electron microscopy (SEM). The freeze-dried starch products were stick on a specimen holder using a silver plate, and then coated with gold in a vacuum evaporator (JEE 400, Jeol). The obtained specimens were viewed in a JEOL JSM 520scanning electron microscope 0 at the accelerating voltage of 10 kV.

3. Results and discussion

3.1. C CP/MAS NMR spectroscopy

Fig. 1 presents ^{13}C CP/MAS spectra of potato starch treated with high pressure at 600 MPa for 2 and 3 min. The chemical shifts of potato starches treated with high pressure showed three major peaks with high-intensity resonances oscilating at 72, 61 and 102 ppm regions. The obtained spectra of pressurised starches seem to correspond, to some extent, to those obtained by Gidley and Bociek (1985) for native potato starch obtained under conditions similar to those of our measurements. Those authors distinguished three peaks with similar intensities compared to the high-intensity resonances observed in the case of pressure-treated starches. They assigned the resonances in the ranges of 60–64 and 94–105 ppm to C-6 and C-1 sites, respectively. The major resonance resolved at the 68–77 ppm region was related to the C-2,-3,-4, and -5 sites. The resonance at the 60–64 ppm region (C-6) was also found to be characterised with higher cross-polarisation, which resulted directly from the presence of the methylene carbon in C-6 sites compared to the other sites related to methine.

Apart from the peaks discussed above, in the ^{13}C CP/MAS spectrum of high pressure-treated starches some other resonances were observed. For starches treated with high pressure for 2 and 3 min it was possible to distinguish freshly appearing resonances at 81.23 and 82.46 ppm, respectively (Fig. 1a and b), characteristic of amorphous starch (Gidley & Bociek, 1985). Furthermore, those authors found almost no signal intensities at this region in the case of the analysis of crystalline material. This may indicate that both peaks, observed in the spectrum of pressurised potato starches, can be assigned to C-4 sites and should be related to the amorphous sites within the starch structure. The spectra of potato starches treated for 2 and 3 min seemed to have different intensity and shape (Fig. 1a and b). In the spectrum starch pressurised for 2 min, the signal corresponding to C-4 carbon (appearing at 81.23 ppm) was characterised by significantly higher resonance intensity

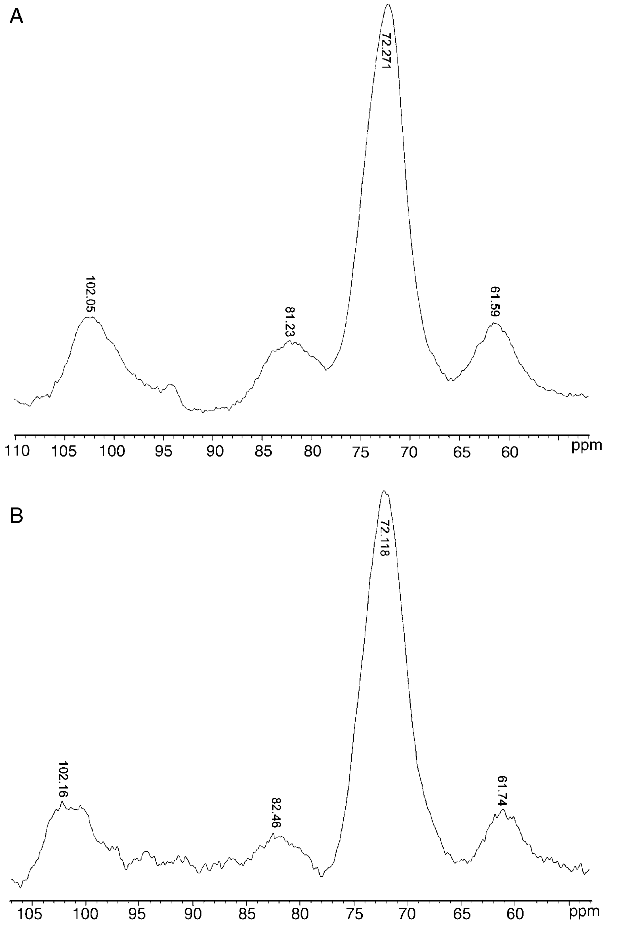

Fig. 1. ^{13}C CP/MAS NMR spectra of potato starch treated with high pressure at 600 MPa for (A) 2, and (B) 3 min.

compared to the spectrum of starch treated for 3 min (appearing at 82.46 ppm), although both resonances were characterised by a slightly different, Gaussian-like shape.

Bogracheva, Wang, and Hedley (2001), studying spectra of crystalline material, found that the shape of the obtained resonances can be typical of ordered or unordered components. They showed that a signal with double C-1 peak at 99–100 ppm region was characteristic for crystalline material. For amorphous material, the smooth and wide peaks for C-1 (102–105 ppm) and C-4 (81–83 ppm) were found by these authors. The difference in the shape observed between crystalline and amorphous structure was ascribed to the formation of double helices by the molecules which—unlike amorphous molecules—are in the strictly determined conformation. In the spectra of potato starches treated with high pressure at 600 MPa for 2 and 3 min (Fig. 1) it was not possible to distinguish any Lorentz-shaped peaks related to an ordered conformation. All the peaks in the analysed spectra, except for that assigned to resonance for C-2, -3 and -5 carbons, were smooth and wide indicating rather amorphous sites within the starch treated with high pressure at the level of 600 MPa.

3.2. F.t-i.r. spectroscopy

The changes in the physico-chemical structure of potato starches treated with high pressure were also confirmed in further analyses. Fourier transform infrared spectra of native and high pressure-treated starches are presented in Figs. 2–4. All the obtained spectra showed major bands in the region of 950–1200 cm^{-1}. It is well known that in this region of F.t.-i.r. spectrum, starch demonstrates bands that are sensitive to the polymer conformation and hydration (Rubens & Heremans, 2000). The spectrum of native potato starch (Fig. 2) showed interesting, from the point of view of

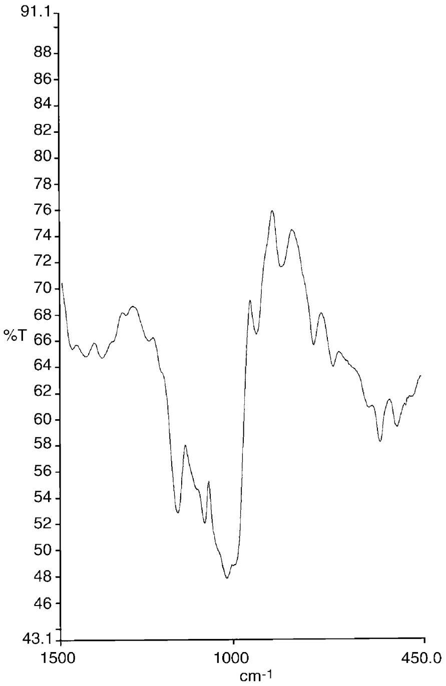

Fig. 3. F.t.-i.r. spectra of potato starch treated with high pressure at 600 MPa for 2 min.

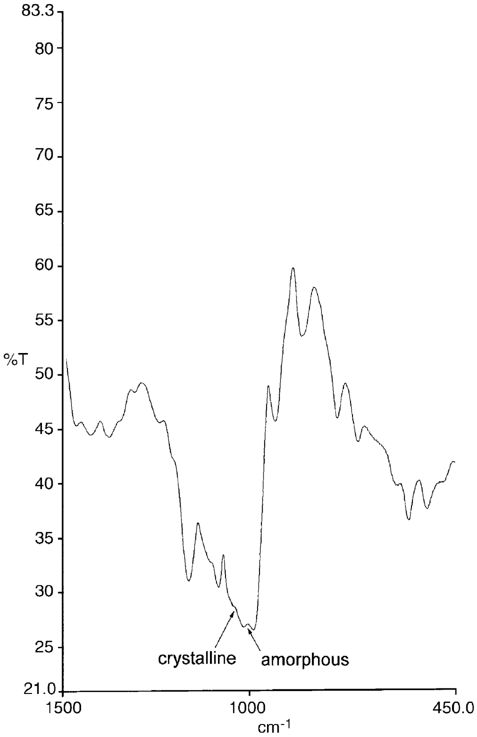

Fig. 2. F.t.-i.r. spectra of native potato starch.

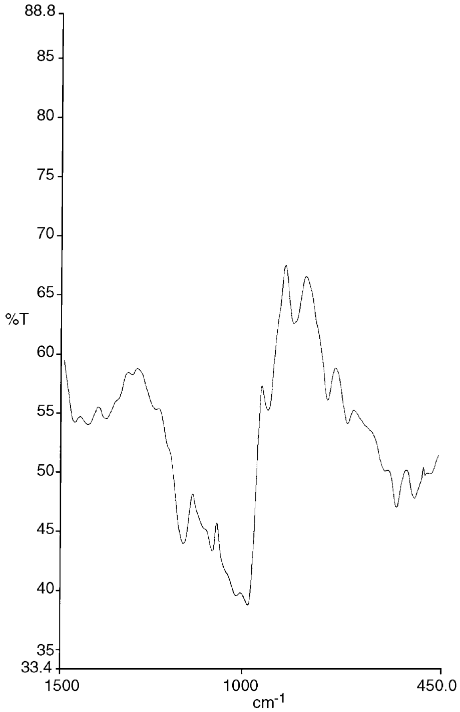

Fig. 4. F.t.-i.r. spectra of potato starch treated with high pressure at 600 MPa for 3 min.

semi-crystalline nature of starch, bands at the 1047 and 1018 and cm^{-1} region. Taking into consideration the results of Smits, Ruhnau, Vliegenthart, and van Soest (1998), it is possible to assign those bands to a more organised and amorphous part of starch, respectively. The other band observed at the 995 cm^{-1} region was found to be sensitive to water (Smits et al., 1998).

The F.t.-i.r. spectra of the high pressure-treated starches (Figs. 3 and 4) showed significant changes mostly in the intensity of the above-mentioned bands related to the time of treatment. Pressurisation of starch at 600 MPa for 2 min was found to cause a substantial increase in the band intensity at the 1018 cm^{-1} region, and its broadening. This phenomenon interfered with a decreasing intensity of a band at the 995 cm^{-1} region. The broadening of the band at 1018 cm^{-1} probably caused overlapping of these two bands and consequently the band at 995 cm^{-1} can be observed as a shoulder of 1018 cm^{-1} one. In contrary, in the case of starch pressurised for 3 min (Fig. 4), the band at the 995 cm^{-1} region seemed to be more evident compared to native starch as well as that pressurised for 2 min. The region assigned to the amorphous part of starch was resolved at 1017 cm^{-1} region with significantly low intensity. After pressurisation of starch at 600 MPa, broadening of the band at the 1047 cm^{-1} region was also observed. According to the suggestion of van Soest, de Wit, Tournois, and Vliegenthart (1994), the changes in the intensities of bands are due to the changes in specific starch conformations, such as long-range ordering and crystallinity. It is possible to state that the wide bands resolved in the spectrum of pressurised starches can be considered indicative of polymer disordering and an increase in the number of conformations. That phenomenon seems to be closely linked to the statement that high pressure induces a reversible hydration of the amorphous phase followed by an irreversible distortion of the crystalline region leading to the alteration of granule structure (Rubens & Heremans, 2000).

3.3. DSC analysis

Calorimetric measurements of gelatinization process of pressure-treated (600 MPa) starches showed a decrease in temperatures (T_o, T_p and T_c) compared to the native sample (Table 1). As the time of the treatment proceeded, the total enthalpy of starches pressurised for 2 and 3 min was observed to decrease substantially. In contrast, the time of treatment had no significant effect on the values of peak temperatures. The DSC results seem to confirm the studies on starch structure and clearly indicate that high pressure at 600 MPa caused some kind of alternation in granule structure. The extent of those changes was represented by a decrease in gelatinization temperatures and an increase in amorphic (disordering) conformations (Figs. 1 and b and 3 and 4). It was reported by Lewandowicz, Jankowski, and Fornal (2000) that changes in some physical properties, i.e. gelatinization temperature, were affected by the alternations of the crystalline structure. According to Rubens and Heremans (2000), high pressure treatment of starch affected melting of the crystalline structure.

Table 1
DSC analysis of native and high pressure-treated potato starch

Sample	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)
Native potato starch				
1	65.11	70.20	77.66	16.58
2	64.35	69.67	76.15	15.17
Mean	65.04	70.08	77.17	15.96
Stand. Dev.	0.83	0.83	1.33	0.94
600 MPa for 2 min				
1	57.23	63.87	70.56	5.90
2	57.70	64.50	71.44	5.19
Mean	57.47	64.19	71.00	5.55
Stand. Dev.	0.33	0.45	0.62	0.50
600 MPa for 3 min				
1	59.33	66.53	73.35	4.43
2	58.24	64.87	71.79	4.18
Mean	58.79	65.70	72.57	4.31
Stand. Dev.	0.77	64.87	1.10	0.18

3.4. SEM study

Scanning electron microscopy analysis confirmed that high pressure altered the starch granule structure (Fig. 5a). SEM pictures showed that, like in native starch, the majority of starch granules treated with high pressure retain granular shape. However, differences in surface characteristics of pressurized granules are evident (Fig. 5b). These observations were closely related to the previous study on high pressure-treated starches (Stute et al., 1996; Stolt et al., 2001). Many granules were characterised by significant deformations, caused by pressure and following freeze drying. In addition, some of them showed clear network-like probably gel structures. All the mentioned changes may also indicate significant changes in the internal structure of the granule. Taken at the same magnification picture of starch granules treated at 600 MPa but for 3 min clearly depicted that the prolonged time of the process is responsible for higher destruction of granule integrity leading to its further destruction (Fig. 5c). The step-by-step effect of high pressure (600 MPa/3 min) on starch granule structure is depicted in details in Fig. 5e–f. Fig. 5d shows the granule with relatively smooth surface which cracked along *y*-axis. This enabled an inside look into the granule structure. The outer part of the granule seemed to be more resistant to any changes showing a very compact condensed layer. In the inner part of the granule, some fibrillar structures are clearly visible. The observed fibrils correspond to semi-crystalline lamellas of the granules, but the changes in the granule structure are always related to the interaction of both starch polymers–crystalline and amorphous. The high pressure-treated granule, when cut, demonstrated two distinct zones of its destruction (Fig. 5e). The outer zone (starch granule envelope) formed by unchanged structures corresponds to

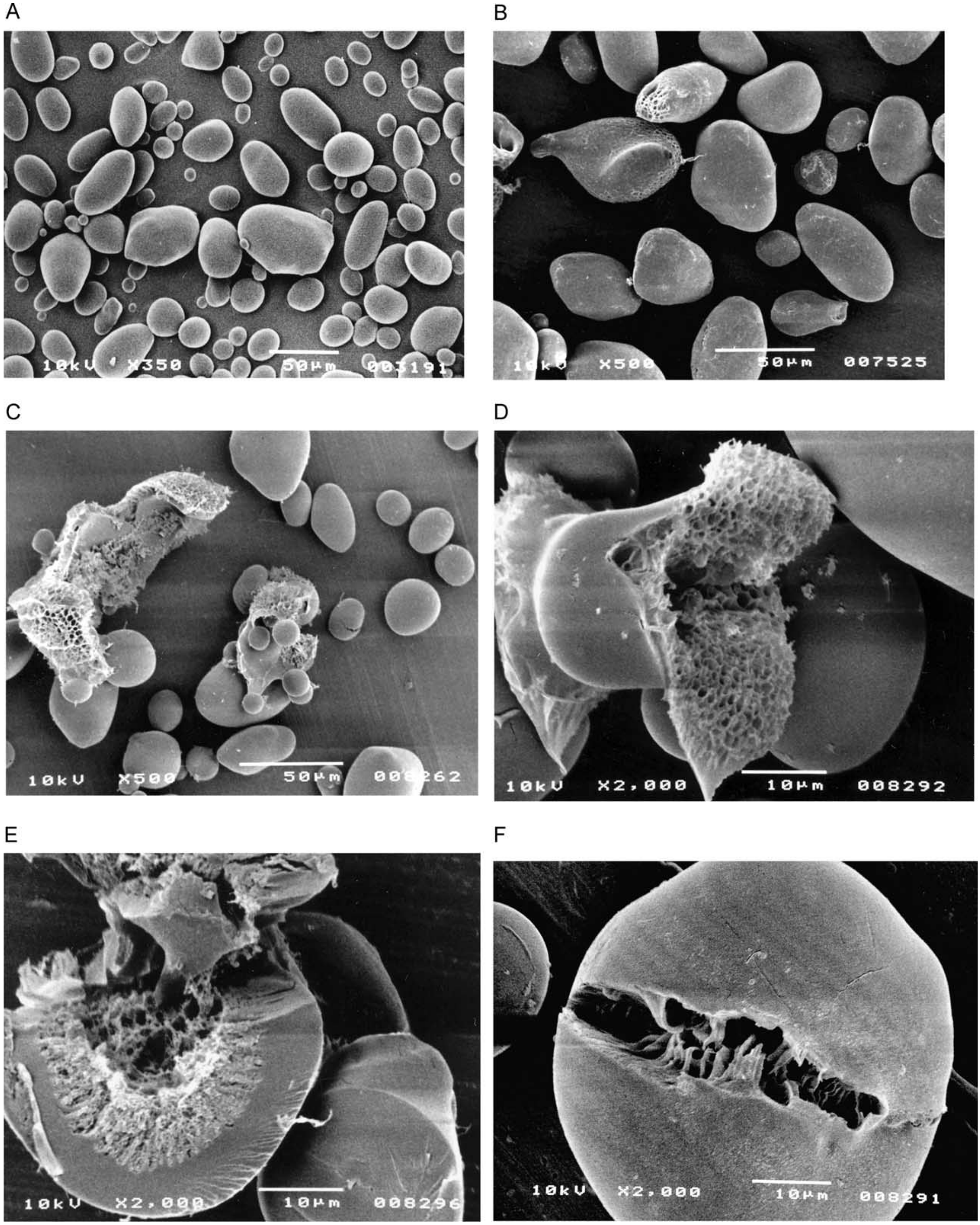

Fig. 5. SEM microstructure of potato starch: native (A); treated with high pressure at 600 MPa for 2 min (B) and 3 min (C); (D)–(F) details of starch structure treated for 3 min.

a more organised (dense) part of the granule. Svergmark and Hermansson (1991) found also that the surface of potato granule is formed by dense material that was resistant to water-heat treatment. Sevenou, Hill, Farhat, and Mitchell (2002) found that the outer structure of potato and amylomaize starch granules was more ordered on their outer regions. This could explain the higher resistance of potato starch to enzyme hydrolysis as well as high pressure treatment (Rubens, Snauwaert, Heremans, & Stute, 1999). Błaszczak et al. (2003) suggested that the external part of starch granule differs significantly from the interior one in its uniformity and is likely to be composed mainly of amylopectin having also a wide range of high molecular mass fragments. In contrast, the inner zone is almost completely filled with gel-like network, with empty spaces growing in diameter towards the centre of the granule. Other, completely open starch granule is shown in Fig. 5f. Regular gel-like structure with the remaining empty spaces after water evaporation during freeze drying is clearly visible, but the granular form of starch is still kept. The gel-like structure formed inside the granule may result from hydration of the amorphous phase and/or melting of the crystalline structures (Rubens & Heremans, 2000).

4. Conclusions

The ^{13}C CP/MAS NMR analysis of potato starch treated with high pressure (600 MPa for 2 and 3 min) indicated resonances at 81.23 and 82.46 ppm corresponding to amorphous sites within starch structure. The pressurisation of starch was found to significantly affect the intensity of bands typical of the amorphous (1018 cm^{-1}) and crystalline (1047 cm^{-1}) part in potato starch structure, irrespective of the time of treatment. High pressure treatment of potato starch resulted in decreased gelatinization temperatures (T_o, T_p, T_c) compared to the native starch. Along with time of the treatment, the total enthalpy of starches pressurised for 2 and 3 min decreased substantially. In contrast, the time of the treatment had no significant impact on the values of peak temperatures. The SEM analysis indicated that the starch granule surface was more resistant to high pressure treatment than the interior part of the granule. Hydration of the amorphous phase and/or melting of the crystalline structures resulted in the formation of gel-like structures inside the granule structure.

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