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The effect of microwave heating of fresh orange peels on the fruit tissue and quality of extracted pectin

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Abstract

The effect of microwave pretreatment of fruit raw material on some physical properties of the orange tissue was investigated. Scanning electron micrographs showed that microwave heating led to destruction of the parenchymal cells. It was also found that specific surface and the water absorption capacity of the orange tissue, and the endogenous enzymes of the peels were inactivated. As a result of this pectin extraction was facilitated. Considerable increasing in pectin yield and was attained (190% from oranges from crop of 1996 and over 250% for oranges of crop 1997 as compared with control). The quality of the pectin was also improved.

Keywords: Orange peels; Microwave heating; Pectin; Pectolytic enzymes

1. Introduction

The texture of fruits and vegetables during growth, ripening and storage depends on the quantity and quality of pectin present (Voragen, Pilnik, Thibault, Alexos, & Renard, 1995). Pectic substances are the most complex compounds from the cellular polysaccharides–cellulose and hemicelluloses. The latter, together with proteins and lignin, are constituents of the cell matrix which is built from the walls of many cells and they form the skeleton of the plant tissue. The changes that take place in the plant tissue during storage and processing affect the composition of pectic substances, too. This is of great significance to their application in the food and flavour industries as additives-gelatinizing agents, thickeners and emulsifiers (Kratchanov, St Stamov, & Pantcheva, 1982; Pilnik & Voragen, 1991).

The use of a suitable method of extraction is of importance during pectin production in order that a good yield of pectin is obtained and its properties are preserved. Pectic polysaccharides are of high molecular weight and closely connected with the other polymer components in the cell walls which inhibits their release from the cell matrix. Therefore, preprocessing of the plant material is often applied to facilitate pectin extraction. The processing methods most often used are enzyme or physical treatments (Thibault, Dedreu, Geraeds, & Rombouts, 1988; Voragen et al., 1995) or physical (Kratchanov, Marev, Kirchev, & Bratanoff, 1986; Manabe, Naohara, Sato, & Okada, 1988; Panchev, Kirchev, & Kratchanov, 1988; Thibault, Ralet, Axelos, & Della Valle, 1996; Osterveld, Beldman, Schols & Voragen, 1996).

In our previous papers it was established that the pretreatment of the fruit material by microwave heating led to a considerable increase in the yield and quality of pectin (Kratchanova, Panchev, Pavlova, & Shtereva, 1994). These results have been confirmed by experimental data obtained by Ilina, Zemskova, Donchenko, & Uvracheva, 2000; Kohg, Lin, & Chen, 2000; Fishman & Chau, 2000; Fishman, Chau, Hoagland, & Aygad, 2000; Fishman, Chau, Coffin, & Hotchkiss, 2003, during investigations into the microwave treatment of citrus peels and apple pomace. We have previous reported (Kratchanova, Pavlova, Panchev, & Kratchanov, 1996) that the benefit were particularly marked during extraction of pectin from orange peels. The influence of the intensity of the electromagnetic field and the duration of microwave heating were studied. It was determined that with an increase of the intensity of the field and the duration, the yield of pectin increased between 180 and 240% compared with the control. At higher field intensities,

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however, prolonging the period of microwave heating led to a decrease in gel strength. That implied that optimization of the microwave heating conditions is needed for each particular case of microwave treatment of the pectin material.

The aim of this paper is to investigate the cause of the increase in pectin yield as a result of microwave heating of fresh orange peels.

2. Materials and methods

Two lots of oranges of the Navel type (crop 1996 and 1997), imported from Greece were used in this study. The peels were removed, then finely cut and processed following two procedures: part of the material was dried in a laboratory drier at 60 °C (control sample), while the remainder was pretreated in a microwave oven and then dried in a laboratory drier at 60 °C.

2.1. Microwave heating of the fresh orange peels

The orange peels (200 g) were processed in glass vessel and heated in microwave oven (Samsung 900) with different duration of exposure: 5, 10 and 15 min and with different power: 0.45, 0.63 and 0.9 kW.

2.2. Extraction of pectin

The dry mass (50 g), obtained after drying 200 g of fresh orange peels, was subjected to extraction by adding 2.5 l water. The pH was adjusted to 1.5 with 0.5 M HCl. The mixture was then heated to 80–82 °C and extraction was carried out with continuous stirring for 1 h. The hot masses were filtered through a cloth. After cooling, the filtrate was coagulated using an equal volume of 96% ethanol and left for an hour. The coagulated pectin was separated by filtration, washed once with 70% acidic ethanol (0.5%HCl), then with 70% ethanol to a neutral pH and finally with 96% ethanol. It was dried at 60 °C in a laboratory drier.

2.3. Scanning electron microscopy

The dried samples were cut into pieces with dimensions approx. 1 mm in depth and up to 5 mm in length. The pieces were glued to a copper holder using silver conducting paint. A 20–50 nm carbon film was then deposited on the orange peel specimens only and on their holders by thermal evaporation in a vacuum evaporator at a pressure $p = 10^{-5}$–10^{-6} Torr. The samples prepared in this way were observed using an electron microscope JEM 1200 EX with a scanning image device ASID 10 at accelerating voltage 20 kV.

2.4. Methods of analysis

The anhydrouronic acid content (GalA) of the initial material was determined by the method of Gee, McComb, and McCready (1958).

Analysis for pectin was implemented by the method of Owens et al. (1952). The statistical deviation of the results was calculated by the method of Brandt (1975). The gel strength was determined by the Tarr–Baker method according to the procedure described by Bender (1949). Intrinsic viscosity $[\eta]$ was calculated according to the Huggins' equation $\eta_{sp}/c = [\eta] + K'_H[\eta]^2 c$ Moravetz (1967). The viscosity average molecular mass M_η was determined by owing the Mark–Houwink relationship reported by Anger and Berth (1986).

Polygalacturonase/PG/and pectinmethylesterase/PME/activities. Two Hundred grams of finely cut fresh orange peels were extracted with 1 l 0.2 M sodium acetate (pH 7.9) as the mixture was homogenized on Polytron homogenizer for 10 min The sample were stored in the refrigerator for 24 h, after which it was centrifuged for 40 min at 6000 × *g*. The supernatant was turbid, increasing is turbidity from the control to the microwave treated samples, being most turbid at 0.93 kW. It was filtered through Glass fiber disc filters type AP above 1 μm (Millipore). Then the supernatant was separated and ammonium sulphate was added to 70% concentration and the precipitated proteins were removed by centrifugation (30 min, 4500 × *g*) and diluted with 5 ml 0.1 M phosphate buffer (pH 7.0). The solution was dialyzed for 48 h against distilled water and after dilution to the required volume with the same phosphate buffer the protein content, polygalacturonase and pectinmethylesterase were determined.

The protein content was determined by the method of Lowry, Rosenbrough, Forr, and Randoll (1951). Polygalacturonase was determined by the viscosimetric method and pectinmethylesterase—according to the titrimetric method Markovic, Kraus, and Slezarik (1980).

The specific surface was determinated by the method of nitrogen porosimetry Gregg and Sing (1967). The apparatus in experiments was a Physical Adsorption Analyser Accusorb 2100, produced by Micromeritics Instrument Corporation, USA.

Water binding capacity of the orange peels was determinated by the traditional weight method (Kabbert et al., 1993).

3. Results and discussions

As can be seen from Table 1, the orange peels differed in polyuronide content and degree of esterification. It is worth noting that the microwave treated samples were had a higher GalA and DE than the control. The increased GalA values can be explained by the improved penetration in the plant tissue of 0.1N NaOH during the titrimetric analysis of the peels, and the increased DE values due to the inactivation of the pectinesterase. This is shown below.

The next series of experiments was dedicated to studying the effect of the intensity of the microwave field and duration of microwave exposure on the yield and quality of

Table 1
Analysis of the dried orange peels

Intensity of microwave heating (kW)	Time (min)	GalA (%)	DE (%)
Navel-1996 control sample		18.0	74.7
0.45[a]	10	18.1	77.1
0.63[a]	10	18.4	77.0
0.90[a]	10	19.4	78.3
Navel-1997 control sample		14.6	70.6
0.63[a]	10	15.1	72.9
0.90[a]	5	15.2	72.9

[a] The samples were dried after Mw-heating.

pectin. Data from oranges of the Navel 1996 and Navel 1997 type (Table 2) confirm our observations in previous studies (Kratchanova et al., 1994, 1986) that there was an inverse correlation between the field power and duration of exposure mainly expressed for 0.45 and 0.63 kW power. In case of a weaker field, longer microwave treatment is needed. Duration of acceptable microwave heating was reduced for the higher values of the field power because of burning of the material. The data in Table 2 show that the increase in the pectin yield as a result of microwave heating also varied with the crop. The yield was about 190% for the orange crop of 1996 and over 250% for crop 1997 as compared with the control.

We made an attempt to explain these results by determining some changes that took place in the plant tissue of orange peels after microwave treatment. As is known, the energy of the super-high frequency (2450 MHz) electromagnetic field is mainly converted into heat in substances made up of polar molecules. As a result of intensive vapor formation in the capillary-porous structure of the plant material, large pressure is built up, modifying its physical properties. These changes were tracked by measuring the changes in the capillary-porous properties of the materials and the change in the activity of pectinmethylesterase contained in orange peels. The changes in the capillary-porous properties of the material were determined by measuring the specific surface and the water absorption capacity. It was found that after a 10-min heating with a field power of 0.45 kW the specific surface of the microwave treated samples increased to 1.87 $m^2 g^{-1}$ as compared with the 0.43 $m^2 g^{-1}$ of the control. The water absorption capacity of the microwave treated materials also rose to 6.15 g/g as compared with the 5.29 g/g of the control. The capillary-porous characteristics of the fruit tissue improved as a result of the increase in the pressure and temperature in the inside of the tissue.

Qualitative confirmation of this fact is given by the photos of the surface of four samples of orange albedo by scanning electronic microscopy presented in Fig. 1. It can be seen that microwave heating led to severing of the parenchymal cells. The damage to the plant tissue increased with the rise in the intensity of the microwave field, which was expressed in increase of the intracellular spaces (scanning electron micrographs 2, 3 and 4). Besides, at 0.90 kW (scanning electron micrograph 4) an increase in the in-depth destruction of the tissue was observed.

It would be of interest to interpret the data on the pectin yield and GalA of the raw materials (Tables 1 and 2). It can be seen that a slight increase in GalA of the raw material causes a considerable increase in the pectin yield. It is obvious that the main reason for the increased yield in the samples subjected to MW-heating was the improved capillary-porous characteristics of the raw material. This ensured better and faster permeation of the extracting agent.

On the other hand, it is known that orange peels contain pectolitic enzymes among which polymetylesterase is especially active. In a previous research of ours

Table 2
Effect of microwave heating of fresh orange peels (1996 and 1997) on pectin yield and characteristics

Intensity of microwave field (kW)	Time of microwave heating (min)	Yield of pectin (g per 100 g dried peels)	GalA (%)	DE (%)	Molecular weight (M_η)	Intrinsic viscosity [η] dl.g^1	Gel strength ^{o}TB
Control sample	1996	9.6	66.4	66.4	54 000	1.9	130
0.45	10	16.6	68.2	71.2	55 000	2.1	152
0.45	15	16.4	68.4	70.6	65 000	3.1	150
0.45	20	17.0	67.1	72.9	72 500	4.5	164
0.63	5	17.2	66.7	73.6	66 000	2.3	168
0.63	10	17.6	68.3	73.5	64 500	3.9	162
0.90	5	17.8	66.6	73.8	69 000	4.2	180
0.90	10	18.0	66.5	74.8	64 000	3.1	180
Control sample	1997	6.0	65.7	65.6	83 500	2.0	109
0.63	5	14.0	69.9	74.2	87 000	2.0	182
0.63	10	15.4	70.1	74.9	82 000	1.9	169
0.63	15	Material bruns					
0.90	5	15.0	68.6	70.4	84 000	1.96	182
0.90	10	Material bruns					

Standard deviation for results of GalA is 0.43% and for the results of DE is 0.11%.

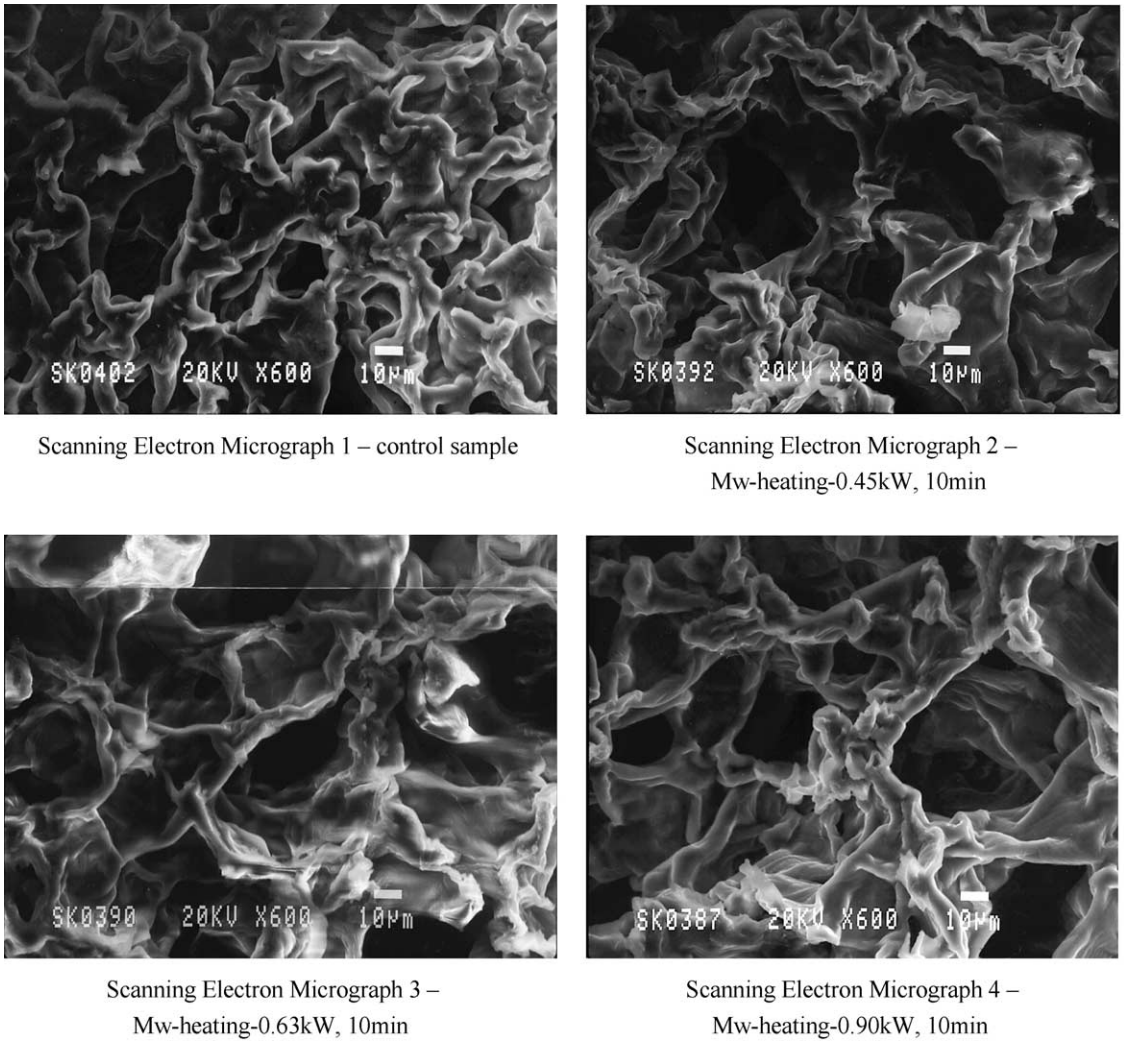

Fig. 1. Scanning electron micrographs of orange peels tissue.

(Kratchanova, Pavlova, Shtereva, & Benemu, 1989) it was established that pectinesterase interacts with the pectic substances in the orange peels and reduces their solubility. This hampers pectin extraction from the fruit plant tissue. The measurement of the pectinesterase activity in the dry orange peels in this study showed that a 10-min heating in a Mw field with a power of 0.45 kW led to a considerable inactivation as compared with the control (Table 3). Further increase in the power of the Mw treatment lead to a complete suppression of the pectolitic activity in the orange peels under study. In this work it is shown that the inactivation of the pectinmethylesterase by Mw treatment leads to an increase extractability of pectin from the material and that the pectins obtained after a microwave pretreatment have a higher degree of esterification, molecular mass, and gel strength.

Table 3
Influence of microwave heating of fresh orange peels (1996) on their soluble protein and enzyme activity

Sample	Orange peels	Enzyme activity		Extracted protein μg/g material	Specific enzyme activity	
		PG [U] γ/g material	PME [U] γ/g material		PG [U] mg prot.	PME [U] mg prot.
1	Control sample	27.2	36.7	920	29.6	39.9
2	Pretreated 10 min 0.45 kW	21.5	0	414	51.9	0
3	Pretreated 15 min 0.45 kW	0	0	400	0	0
4	Pretreated 10 min 0.63 kW	0	0	301	0	0
5	Pretreated 10 min 0.93 kW	0	0	240	0	0

4. Conclusion

Microwave pretreatment of fresh orange peels led to destructive changes in the plant tissue. The changes resulted in an increase in the capillary-porous characteristics and the water absorption capacity of the plant material. The heating inactivated the pectinesterase activity in the oranges. These changes in the plant tissue after a microwave pretreatment gave an opportunity for (or rather they cause) the considerable increase in the yield of extractable pectin and improvement of its parameters (e.g. DE, molecular mass, and gel strength).

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