ELSEVIER

0144-8617(94)00048-4

Carbohydrate Polymers 25 (1994) 141–144

0144-8617/94/$07.00

Extraction of pectin from fruit materials pretreated in an electromagnetic field of super-high frequency

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(Received 28 January 1994; revised version received 14 July 1994; accepted 22 July 1994)

Laboratory studies on the extraction of pectin from orange, lemon and apple wastes pretreated in an electromagnetic field of super-high frequency were carried out. A 10-min preliminary microwave heating (2450 MHz, 0·5 kW) of crushed fruit materials was found to provide a higher pectin yield. The extracted pectin exhibited higher values for degree of esterification and gel strength compared with the control sample. The effect depends on the kind of raw material.

The favourable effect of microwave heating on the yield and quality of pectin is assumed to be due first to the partial disintegration of the plant tissue and hydrolysis of protopectin, and second, to the rapid inactivation of the pectolytic enzymes in the raw material. The second hypothesis has been partly confirmed by model experiments.

The results presented can serve as a basis for improving pectin manufacturing. It is expedient that fresh pectinous raw materials should be subjected to microwave heating before drying.

INTRODUCTION

Pectic substances are widely used in the manufacturing of foods as gelling agents, thickeners and stabilizers of dispersed systems (Pilnik & Voragen, 1980, 1992). Their main source are fruit wastes from juice production (apples, lemons and oranges). The yield and quality of pectin depends strongly on the preliminary treatment of the raw material. Various physical and chemical methods have been applied, leading mainly to disintegration of the plant tissue and inactivation of pectolytic enzymes (Kratchanov *et al.*, 1986; King, 1987; Panchev *et al.*, 1988; Fujio & Furuda, 1989).

New physical methods of drying and processing of fruit materials in the food industry have recently been applied. The capacity of an electromagnetic field of super-high frequency (EMF-SHF) to penetrate raw materials and simultaneously heat their total volume and to enhance the heat- and mass-exchange processes, makes the method of microwave heating extremely attractive for drying of fruit and vegetable raw materials (Decareau, 1985; Mudgett, 1989; Bouraoui *et al.*, 1993). It is of interest to apply this method in treating fruit materials designed for pectin extraction. In general, these are juice production waste products (pressings, peels, etc.) and the immediate treatment of this raw material is of uppermost importance for its storage. The effect of the pretreatment of raw materials by EMF-SHF on the quality of pectins obtained has not been described.

In a preliminary study, Manabe and coworkers (1987, 1988) reported the use of microwave heating in the pectin extraction process. This resulted in an increase of pectin yield. Viscosity data for the pectins obtained showed a decrease in the relative viscosity. Data on the gel strength of the pectins were not presented.

We now report a study of the possibility of application of EMF-SHF to the processing of fruit waste from juice production used for the production of pectin, and the effect of this treatment on the quality of the extracted pectin.

MATERIALS AND METHODS

Citrus fruits such as lemons and oranges imported from Greece and apple fruits gathered on the day of study were treated.

Citrus peels after removal and cutting were used for the extraction of pectin. Apple fruits were grated and pressed using cloth. Pressings were used for the production of pectin. Part of the material so prepared was immediately subjected to extraction. Another part was dried at 60°C in a laboratory drying-oven. A third part of the material was treated by EMF-SHF. After microwave heating, the material was divided into two parts. One was immediately subjected to extraction while the other was first dried at 60°C in a laboratory drying-oven and after that extracted.

Treatment of the fruit materials by microwave heating

Fresh orange or lemon peels or apple pressings (200 g) were placed in a glass vessel and heated for 10 min in a household microwave over, type 'Elektronika', power 0·5 kW, at an operating frequency of 2450 MHz.

Extraction of pectin from fresh fruit materials

Fresh orange or lemon peels or apple pressings (200 g) were soaked with 1·8 dm^3 of water. The pH was adjusted to 1·5 using 0·5 N hydrochloric acid. The mixture was heated to 80–82°C and the extraction was completed by continuous stirring for 1 h in a laboratory stirrer MM 24 (Czechoslovakia). The hot mass was filtered through cloth. After cooling, the filtrate was coagulated using an equal volume of 96% ethanol and left for 1 h. The coagulated pectin was separated by cloth filtration, washed only once with 70% hydrochloric acid–ethanol, followed by 70% ethanol to neutral reaction and finally with 96% ethanol. Drying was achieved at 50°C in a laboratory drying-oven.

Extraction of pectin from initial fresh fruit material treated by microwave heating

Fresh fruit material (200 g), such as orange, lemon peels or apple pressings, were placed in a glass vessel and subjected to microwave heating. After cooling, the sample mass was brought to the initial weight value by adding water and then subjected to extraction as described above.

Extraction of pectin from dried fruit material

The dry mass obtained after drying of 200 g of fresh fruit material was subjected to extraction by adding water to the citrus material or apple pressings at ratios of 1:50 and 1:15, respectively (50 g of dry orange peels plus 2·5 dm^3 of water; 45 g of dry lemon peels plus 2·5 dm^3 of water; 55 g of dry apple pressings plus 0·82 dm^3 of water). The extraction was achieved as above.

The same procedure of extraction was applied to the fruit material pretreated by microwave heating and dried in a laboratory drying-oven.

Methods of analysis

The anhydrouronic acid content (AUAC) of the initial material was determined by the method of Gee *et al.* (1958). The analysis for the pectin was carried out titrimetrically after the neutralization method of Owens *et al.* (1952). The gel strength (GS) was determined using the Tarr–Baker method, according to the procedure described by Bender (1949). Isolation of pectinesterase from fresh orange peels and determination of its activity (PEA) were performed by the method described by Kertesz (1955).

RESULTS AND DISCUSSION

The effect of the microwave heating on the fresh fruit material was studied at first. The analysis of the intial fruit material (Table 1) showed that the AUAC of the material was retained, even slightly increased, after treatment by microwave heating, which should be attributed to enhancing the degree of penetration of reagents in the fruit tissue, particularly in the case of the citrus materials. The degree of esterification (DE) decreased after all types of treatment of the fresh materials. A more significant decrease in DE was observed during drying of untreated materials in the laboratory drying-oven at 60°C. This could be ascribed to the fact that the slow increase in temperature activated the fruit pectinesterase while its denaturation proceeded more slowly. These changes took place to a significantly lesser extent during the microwave treatment of the fruit raw materials. In the latter case, heating occurs rapidly in the whole bulk of the material and thus inactivates the pectolitic enzymes. Thus, the

Table 1. Analysis of initial materials

Kind of initial materials	AUAC %	DE %
Orange peels		
Fresh	4·1	78·6
Dry	17·2	76·0
Dried after 10-min microwave heating	17·7	77·1
Lemon peels		
Fresh	3·8	78·0
Dry	17·5	74·5
Dried after 10-min microwave heating	19·1	76·2
Apple pressings		
Fresh	3·6	84·8
Dry	14·4	81·4
Dried after 10-min microwave heating	14·4	84·0

Table 2. Yield and characteristics of pectin of orange peels

Sample No.	Kind of initial material for pectin extraction	Yield of pectin, g per 1 kg fresh material	AUAC %	DE %	Gel-strength, °TB	Yield of 100° pectin, g per 1 kg fresh material
1	Fresh material – Control sample	33·6	70·5	70·5	155	52·1
2	Treated by microwave heating	43·2	70·0	71·5	185	79·9
3	Dried material – Control sample	29·9	73·4	68·9	153	45·7
4	Dried after microwave heating	47·1	70·5	71·3	197	92·8

Table 3. Yield and characteristics of pectin of lemon peels

Sample No.	Kind of initial material for pectin extraction	Yield of pectin, g per 1 kg fresh material	AUAC %	DE %	Gel-strength, °TB	Yield of 100° pectin, g per 1 kg fresh material
1	Fresh material	25·0	75·8	73·0	155	38·8
2	Treated by microwave heating	27·0	78·8	74·3	167	45·1
3	Dried material	19·8	76·1	71·3	149	29·5
4	Treated by microwave heating and dried	27·3	71·1	74·8	237	64·7

microwave treatment of the fresh fruit material exerted a favourable influence with regard to the qualities of the pectic substances in the raw material.

The effect of the preliminary microwave treatment of fresh fruit materials on the yield of the extracted pectin and is properties are presented in Table 2, 3 and 4 along with comparative data on the yield of pectin from untreated materials. For better comparison of the results obtained from fresh and dried materials, the yield of pectin has been expressed per kilogram of fresh material.

In the case of orange peels (Table 2), a higher yield of pectin was obtained from plant material pretreated by microwave heating (samples 2 and 4). It is noteworthy that the highest yield of pectin was obtained from the sample treated by EMF-SHF and dried after that (sample 4). This observation is of high significance for pectin technology. No differences in AUAC of the pectins obtained were observed except for the pectin extracted from directly dried orange peels (sample 3). The degree of esterification was higher in pectins obtained from materials pretreated by microwave heating (samples 2 and 4). The gel strength was considerably higher in pectins extracted from materials pretreated by microwave heating, the highest value found in sample 4 (Table 2).

In the case of lemon peels as pectin source (Table 3), the highest yield of pectin was again observed for materials pretreated by microwave heating (samples 2 and 4). These pectins also showed the highest values of DE and GS, pectin sample 4 showing the highest values.

The same behaviour was observed with apple pressings (Table 4). It should be noted that the difference in the yields of pectin from treated materials and the control samples (untreated by microwave heating) was not great compared with that of the orange and lemon peel samples. The effect of microwave heating is strongly expressed in the GS of apple pectin.

The data on the DE showed that the pretreatment of fresh fruit materials by microwave heating ensured preservation of the DE of the pectins obtained. This could be ascribed to the rapid inactivation of fruit pectinesterase contained in the raw material. This hypothesis has been proved and confirmed by the following test with orange peels: the protein (amounting to 0·05% of the raw material) extracted from untreated (with microwave heating) fruit material showed PEA, while the protein (0·02%), extracted from the treated (with microwave heating) material did not show any measurable PEA.

Another important result is that microwave heating ensured an increase of the pectin yield in comparison with the control sample. That could be attributed to the specific action of the nongradient heating method leading to partial disintegration of the plant tissue, facilitating the penetration of the extracting agent as well as the partial hydrolysis of the protopectin. These effects were specific for the kinds of fruit tissue tested. For this reason the results obtained with various materials were different, but in all cases the microwave treatment favourably affected pectin extraction.

Special attention should be paid to the fact that the yield of pectin from dried pressings increased markedly when these were subjected to microwave heating prior to drying (compare data on the yield from samples 3 and 4 in Tables 2, 3 and 4). It is of prime importance for the production of citrus pectin. This refers to a lesser

Table 4. Yield and characteristics of pectin of apple pressings

Sample No.	Kind of initial material for pectin extraction	Yield of pectin, g per 1 kg fresh material	AUAC %	DE %	Gel-strength, °TB	Yield of 100° pectin, g per 1 kg fresh material
1	Fresh material–Control sample	26·0	61·6	76·7	134	34·8
2	Treated by microwave heating	29·0	59·3	77·3	189	54·8
3	Dried material–Control sample	28·0	67·4	76·5	170	47·6
4	Treated by microwave heating and dried	30·7	69·5	76·9	230	70·6

degree to the production of apple pectin, but again the effect is significant with respect to practical application.

CONCLUSIONS

Based on the laboratory tests on the application of microwave heating to the treatment of fruit materials used for extraction of pectin, the following conclusions could be drawn:

1. The pretreatment of fresh fruit raw materials using microwave heating for 10 min ensured a better extraction of pectin, which resulted in an increase in the yield of pectin: 30–50% for orange peels, 10–25% for lemon peels and 10–12% for apple pressings. This could be attributed to the specific action of the nongradient heating method leading to partial disintegration of the plant tissue as well as the partial hydrolysis of the protopectin.
2. The same pretreatment of the fresh fruit raw materials ensured retention of the DE of the isolated pectin. This effect is due to inactivation of pectinesterase and is more strongly expressed in citrus materials.
3. The GS of pectins obtained from fruit materials pretreated by microwave heating was found to considerably exceed that of control pectin samples from untreated materials.

These results are of considerable importance for pectin production. It is expedient that fresh pectinous raw materials should be subjected to microwave heating before drying.

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