UNIVERSITY OF SZEGED
FACULTY OF ENGINEERING

# REVIEW OF FACULTY OF ENGINEERING

*Analecta Technica Szegedinensia*

SZEGED
2008.

**PUBLISHER:**

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Norma Nyomdász Kft. Kiadó és Nyomda
6800 Hódmezővásárhely, Rárósi u. 10.

ISSN 1788-6392
UNIVERSITY OF SZEGED FACULTY OF ENGINEERING
H-6724 Szeged, Mars tér 7.
Phone: +36 (62 )546 000

## CONTENTS

# PECTIN EXTRACTION FROM BLACKCURRANT PRESS CAKE

*Nóra Pap[2], Sándor Beszédes[1], Szabolcs Kertész[1], Zsuzsanna László[1], Eva Pongrácz[2], Riitta L. Keiski[2], Gábor Szabó[1] and Cecilia Hodúr[1]*

[1]University of Szeged, Faculty of Engineering, Department of Technical and Process Engineering, Moszkvai krt. 5-7, HU-6725 Szeged

[2]University of Oulu, Department of Process and Environmental Engineering, Mass and Heat Transfer Process Laboratory, P.O.Box 4300, FIN-90014 University of Oulu

**Keywords:** pectin, blackcurrant press cake, solvent extraction, microwave-assisted extraction, recovery

## ABSTRACT

The efficiency of microwave activation process in the extraction of pectin from blackcurrant press cake was investigated in this study. Conventionally, pectin extraction is a time-consuming and energy-intensive process. In our experiments, microwave enhanced extraction (MAE) method was used. The applied specific power levels were 5, 10, 15 and 25 W/g, and the processing time applied was between 10-40 minutes. As a control method, conventional hot water extraction (at 80-90°C) was used. The effect of pH on pectin yield was also investigated in both cases. The pectin content was measured by spectrophotometer at 520 nm by the m-hydroxydiphenyl method and was expressed as galacturonic acid equivalent.

The effect of time, pH of the solvent, solid-liquid ratio and the specific microwave power level on the extractable pectin content have been studied. The experiments demonstrated that MAE reduced the extraction time from 9 h to 30 minutes, while the yield of pectin increased. It was concluded that MAE is a viable method for the recovery of valuable compounds from blackcurrant juice processing by-product.

## 1. INTRODUCTION

Natural pectin is a structural unit of fresh cell and a junction between the cells advanced land plants. It exists between the cell walls and its function is to agglutinate the cells to form a compact junction. The pectin consists of a-D-galacturonic acid components, which are partially esterified with methyl alcohol at carboxylic acid end. In industrial applications, pectin is widely used in food and pharmaceutical industry as gel-forming and texturizing agent.

The most used raw materials of commercial pectin extraction processes are apple, orange, sugar beet, berries (e.g. blackcurrant). I the case of the latter, the by-product of blackcurrant juice pressing, contains a great deal of valuable components such as pectin, which are worthwhile to recover.

The commercial pectin extraction processes are based on degradation by acid and deposition. These processes are a very time consuming, taking from 1 to 12 h, and have a large liquid phase demand. In these processes an acidic solution is used, by applying sulphuric, phosphoric, nitric, acetic or hydrochloric acid, and a temperature range from 80 to 100 °C. These conditions may also result in protein degradation and, therefore, they can negatively affect both the quantity and the quality of extracted pectin. These reasons have led to the application of improved, rapid extraction processes, such as microwave assisted extraction (MAE) (Manabe et al., 1988).

In recent years, MAE attracted a growing interest, as it allows rapid extractions of solutes from solid matrices, with extraction efficiency comparable to that of classical techniques. In this type of extraction, microwave energy is used to heat solvents in contact with samples to extract valuable and soluble compounds from the sample into the extractant, such as pectin from apple pomace (Wang et al., 2007), lime (Marshall et al., 2006) and orange peels (Zhongdong et al., 2005 and Kratchanova et al. 2004).

The microwave energy is a non-ionising radiation (frequency 300-300000 MHz) that causes molecular motion by migration of solvent ions in electromagnetic field and rotation of dipoles of polar molecules, such as water (Jones et al., 2002). The efficiency of microwave energy is dependent on the dielectric properties of solvent and the sample. As strongly polar solvent, water can efficiently absorb the microwave energy and transform it into thermal energy, leading to rapid heating of the sample.

Microwaves heat the sample without heating the vessel, therefore the solution temperature rapidly increases leading to a very short extraction time. During microwave irradiation the cells are thermally stressed, the temperature and pressure in the cell will exceed the maximum and the cell walls are ruptured. Thus the skin tissues are opened up by the microwaves more rapidly and extensively than in the conventional hot water method (Zhongdong et al., 2005).

In our work, we investigated the effect of extraction time, pH, liquid to solid ratio and the specific microwave power on the yield of pectin from blackcurrant press cakes by MAE, and compared the MAE method to the conventional hot water and acidic hot water extraction methods.

## 2. MATERIALS AND METHODS

The moisture content of the blackcurrant press cake was 64.5%. The moisture content was determined by drying 10 g of sample at 105 °C for 24 h in a drying cabinet.

For the conventional extraction, an Armfield (Hampshire, Great Britain) pilot solvent extractor was used. The solvent was water at 80 °C, with pH 6.18 without any adjustment, and water adjusted with aqueous hydrochloric acid solution to pH 2. The quantity of sample was 100 g and the solid-liquid ratio was 1:40.

For MAE a single-mode cavity resonator was used, at a frequency of 2.45 GHz. The microwave power of the magnetron is continuously adjustable between 100 and 700 W. The treatments were carried out in a covered PTFE sample holder to prevent evaporation during the irradiation without pressure increase. Temperatures were measured with an infrared thermometer.

The specific microwave power level was changeable by varying the power of the magnetron and using different sample quantities. The specific power levels applied were 5, 10, 15 and 25 W/g wet weight, and the experiments were carried out during 10 to 40 minutes. The solid-liquid ratio varied between 1:5 – 1:20.

The extracted pectin content was measured photometrically at 520 nm by the *m*-hydroxydiphenyl method adapted from Ibarz et al. (2006), and expressed in GA units. A standard curve absorbance/concentration was fitted with the absorbance values at 525 nm from different series of D-galacturonic acid concentration solution. A blank tube with no D-galacturonic acid content was also prepared. The pectin sample was obtained from the blackcurrant extracts, and the water-soluble pectin (WSP) was determined as follows. A sample of 0.1 mL was added to a 15 mL assay tube. In the case of the WSP 0.5 mL of extract and 0.5 mL of distilled water were added followed by the addition of 5 mL of the sulphuric tetraborate solution and placed in a ice-water bath. The mixture was shaken in a vortex mixer to homogenize the sulphuric solution. The cold bath prevents excessive warming, allowing a higher control of the time of the hot sulphuric reaction.

The tube is then placed in a water bath of 80 ± 0.5 °C, for 6 min and then immediately placed in the water-ice bath until reaching room temperature. Further, 0.1 mL of m-hydroxydiphenyl solution was added and, after a vortex stirring to homogenise the mixture, spectrophotometric absorbance at 520 nm was carried out, with absorbance readings being taken at different times.

In some cases, the pectin content was precipitated with isopropanol; a 50-ml sample was washed with 2×150 mL isopropanol, and the precipitated gel was filtered off, dried for 24 h at 30 °C, and then measured by spectrophotometer. All analyses were performed in triplicate.

The energy demand of extraction processes was calculated from the power of magnetron and the filament of the extractor with the following formulas:

$$Q_{extraction} = P_{max} t_{heating} + P_{heater} t_{extraction} \quad [1]$$

$$Q_{microvawe} = P_{magnetron} t_{extraction} \quad [2]$$

## 3. RESULTS AND DISCUSSION

Firstly, commercial hot water and acidic hot water extraction was investigated for the extraction of pectin residues, and the pectin content of the extracts was determined. The results of the extracted water-soluble pectin content for 8 hours' duration of extraction are depicted in Figure 1.

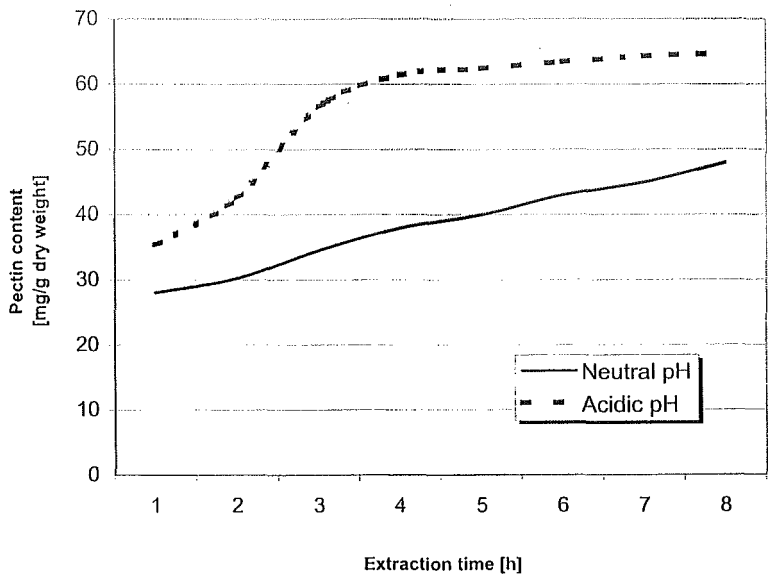


*Figure 1. Pectin content of the extracts in hot water and acidic hot water extraction processes*

Figure 1 illustrates that there was no saturation value in the conventional hot water extraction during the 8 hours of treatment. The connection between the extraction time and pectin yield was linear during the examined interval. When hot water adjusted with hydrochloric acid to pH 2 was applied for the extraction, the pectin yield showed an increasing tendency in the first 4 hours of extraction time, where the curve reached a saturation point.

In the next series of our measurements, the pectin yield from black currant press cake was investigated by applying microwave assisted extraction process. The effects of microwave power level, solid to liquid ratio and the pH of solution were investigated.

Figure 2 illustrates the effect of specific microwave power levels on pectin extraction. The applied specific microwave power was between 5 - 25 W/g changed by adjusting the power of magnetron.

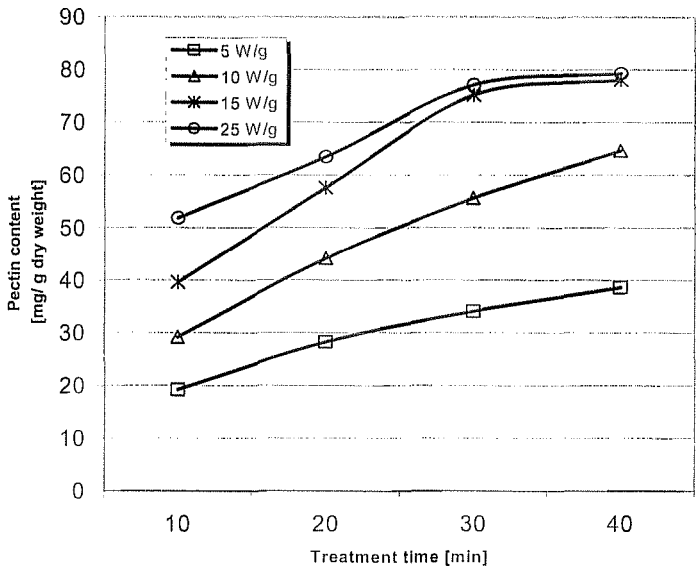


*Figure 2. Influence of specific microwave power on extracted pectin content (solid to liquid ratio 1:10)*

Pectin yield increased with treatment time and increasing microwave power. After the 20 minutes treatment a linear trend was measured when the applied specific power level was 5W/g and 10W/g. At 15 and 25 W/g specific microwave power levels, a saturation value in the pectin yield was reached after 30 minutes. The benefit of the application of higher specific microwave power level was an intensification effect in the beginning of extraction. However, since after 30 minutes the extracted pectin content was the same at 15 and 25 W/g, it was concluded that, further, the maximum power of 15 W/g is to be used.

In another series of experiments, the influence of solvent to solid ratio on the pectin content of the extracts was examined. MAE was carried out using water as a solvent with 5:1, 10:1 and 20:1 solvent to solid ratio. Because of the low pectin yield at 5 W/g microwave extraction and the similarity of the values of pectin content at 15 and 25 W/g the results of 10 and 15 W/g MAE were illustrated on Figures 3 and 4.

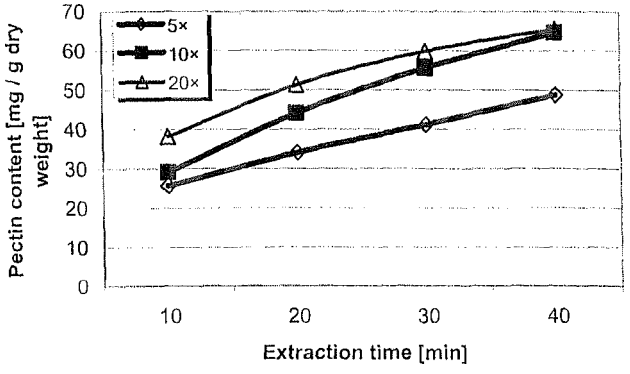


*Figure 3. The influence of solvent to solid ratio on the yield of extraction at 10 W/g microwave power level*

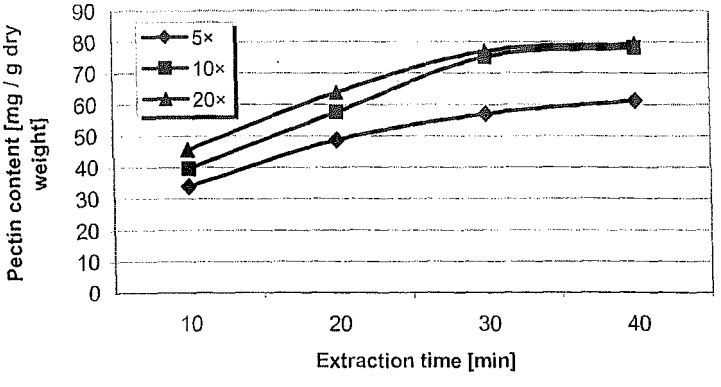


*Figure 4. The influence of liquid to solid ratio on yield of extraction at 15W/g microwave power level*

As Figures 3 and 4 indicate, the higher liquid to solid ratio the higher the extracted pectin was. Higher extraction level was reached with 15 W/g, however, after 30 minutes of irradiation there was no significant yield difference between tenfold and twenty fold dilutions. Therefore, it was deduced that the 10:1 solvent to solid ratio is optimal.

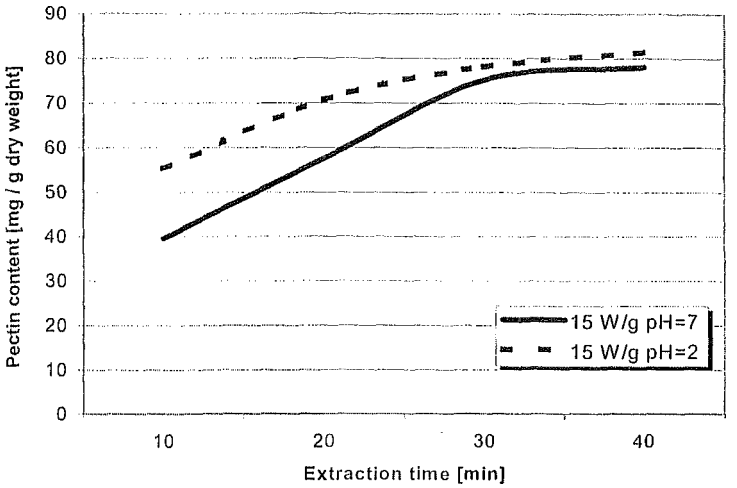


*Figure 5. The influence of pH of the solvent on pectin yield at 15W/g power (liquid to solid ratio 10:1)*

Finally, the influence of the pH of solvent on the extracted pectin yield was investigated at 15 W/g power level and liquid to solid ratio was 10:1. The pH of the solvent was adjusted either to pH 2 or to pH 7. The results are illustrated in Figure 5.

As Figure 5 illustrates, at pH 2 the pectin yield was higher, however, after 30 minutes, there was no significant difference between the amounts of extracted pectin at as compared to the solvent sample at pH 7. However, since the structure of pectin can be easily damaged at low pH, we concluded that the extractions are better to be carried out at pH 7. In this way the use of auxiliary materials can also be avoided.

Finally, to evaluate the efficiency of different extraction methods, the energy demands were calculated. The results are depicted in Figure 6.

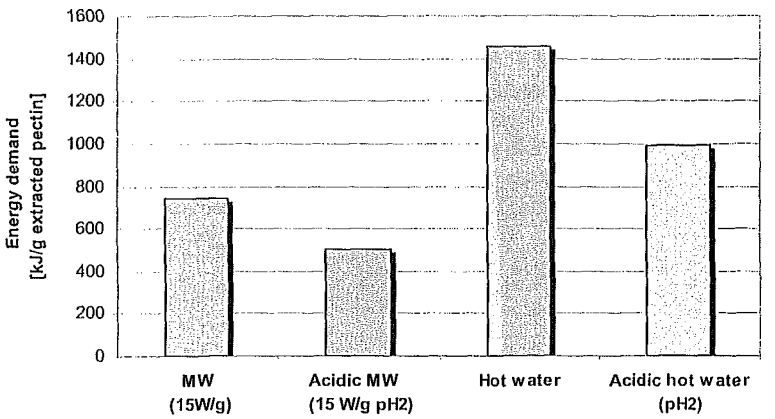


*Figure 6. Energy demand of different extraction methods*

From the aspect of energy demand, MAE is clearly less energy-intensive when compared to hot water extraction. In both extraction methods, lowering the pH of the solvent decreased the energy demand of the process. Notwithstanding, the extraction at pH 7 is recommended, when using MAE, the addition of acid can be avoided, as the energy saving is not so substantial, and at neutral pH the pectin quality is expected to be better.

## 4. CONCLUSION

Microwave heating accelerated the extraction of pectin from blackcurrant press cake. Our experiments show that, as compared to hot water extraction, microwave assisted extraction reduced the processing time from 6-8 h to 30 min, the pectin yield was higher and, the liquid phase demand could be lowered.

It can be concluded that the best operational parameters of pectin recovery from blackcurrant press cake by MAE are 15W/g specific microwave power level, 30 minutes extraction time, 10:1 solvent: solid ratio and pH 7.

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