The Development of a Pectin Extraction Technology for Pea Hulls to Expand the Raw Material Base for the Production of Functional Foods in Russia

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Abstract. Pectin is one of the vital products on the global market due to the increased demand for natural and functional ingredients. The prospective growth of pectin demand from food manufacturers in Russia exceeds the current consumption by manifold because products based on it can be used for disease treatment and prevention. Therefore, it is necessary to restore the production of pectin from Russian-made raw materials. Secondary materials are a promising resource for pectin extraction. There is a scientifically justified and developed pectin extraction technology based on pea grain hulls from pea varieties cultivated at the Federal Research Center for Leguminous and Cereal Crops (Oryol, Russia). The pectin extraction conditions were selected taking into account the physical and morphological properties of pea grain hulls. The implementation of this technology will allow obtaining high-quality pectins that can be used as a gelling component in the production of functional jelly foods.

1. Introduction

According to various sources, the consumption of pectin in the Russian Federation is between 12 and 15 thousand tons a year [1], and 100% of it is imported[2]. China is the leading supplier for the Russian market. It provides 22.1% of the pectin, which is more than some large corporations like CP Kelco, Herbstreith & Fox, and Cargill [3].

The prospective growth of the demand for pectin exceeds the current consumption by several times because products based on it can be used for the treatment and prevention of some diseases among people working in hazardous industries or living in regions with bad ecology [4, 5, 6].

The pectin used in healthy nutrition is natural and non-modified, unlike the commercial modified pectin used in the food industry which does not have positive effects on the human body [1].

Therefore, it is necessary to restore the production of pectin from Russian-made raw materials.

2. Relevance

Pectin is one of the vital products on the global market because consumers tend to reject artificial food supplements and genetically modified products, while food manufacturers tend to opt for natural and functional ingredients.

Pectins were not produced in Russia for a long time because there were not enough raw materials and there were no technologies to arrange profitable production.
The analysis shows a great demand for pectic substances from commercially successful food manufacturers, pharmaceutical companies, and nutritional supplement producers [7, 8].

The food industry is the main consumer of pectin which is used as a gelling agent and a thickener there (Figure 1).

Over recent years, Russia has been developing technologies and projects aimed at solving the problems of pectin production. Togliatti Special Economic Zone is planning to implement an investment project that relies on sugarbeet pulp, apple pomace, sunflower heads, and topinambur as the raw materials [9]. In the Udmurt Republic, Ekopektin LLC has launched the construction of a plant to produce liquid pectin using a unique eco-friendly technology [10]. The Optovo-Raspredelitelny Centr LLC, an investment for the project in Leningrad Oblast, is planning to produce pectin using the technology developed by Kuban State Agrarian University [11].

The project for the Development Strategy for Food and Processing Industries in the Russian Federation until 2030 specifies the requirement to develop the raw material base for the industries, introduce advanced processing, and use production wastes to improve the products per a unit of raw material processed [12].

Therefore, searching for the sources of raw materials containing pectins and developing pectin extraction technologies for them is a very promising area of research.

The main raw materials for industrial pectin production include citrus fruit and apple pulp. There are also pectin extraction technologies that can use pumpkins, oriental radishes, kiwi, and other crops. Secondary raw material sources (cotton, nut, and bean shells, seed hulls, conifer-tree bark, berry pulp, etc) are a promising kind of raw materials for pectin extraction [13, 14, 15, 16, 17, 18].

3. Formulation of the problem
The development of a pectin substance extraction technology for pea grain hulls will ensure zero-waste processing of secondary resources (pea grain hulls) that were not used previously and produce high-quality pectin along with extra insoluble dietary fiber.

The implementation of this technology will result in solving the problem of production waste disposal and expanding the raw material base for the production of functional foods.

4. Theoretical part
The pea varieties Temp and Amior (wrinkled seeds) cultivated by the Federal Legume and Cereal Research Center (Oryol, Russia) were used as the raw material for the development of a pectin extraction technology from pea grain hulls. The content of hulls for the pea samples in question was 7.1% (Temp) and 10.5% (Amior).

Pea grain hulls consist of the rind, whose outer wall is covered in aquitard cuticle. Then there is
the hypoderm and several layers of pap. Pectin is localized in the walls of the pap tissue. Thus, to improve the extraction of pectin substances, the hulls must be pulpified [19]. The highest yield of pectin substances was obtained when the prepared pea grain hulls were reduced to particles of up to 1 mm in size.

Depending on the material, the extraction of pectin substances can be done using organic (citric, ethane, and acetic) and mineral (hydrochloric) acids of various concentrations, as well as milk whey, ammonium oxalate, enzymic products, etc.

Experiments showed that the mineral acid virtually destroyed the shells of the cotton pods [20]. Therefore, we used the solution of hydrochloric acid as a hydrolyzing agent as the most active substance [21].

When experimenting with water solutions of hydrochloric acid of various concentrations to hydrolyze-extract pectins from pea grain hulls, we established that the best concentration for this process is 2.0% because it ensures the highest yield of pectin substances from the materials. The reduction of acidity leads to the reduction of the yield of pectin [22].

5. Results of experimental studies

The hydrolysis extraction of pectin substances from pea grain hulls was carried out by applying a 2.0% water solution of hydrochloric acid with the irrigation module of 1:7, the ambient pH between 1.2 and 1.5, the temperature between 75 and 78°C for 110-120 minutes. Under these conditions, the yield of pectins from pea grain hulls is at its highest.

Increasing the temperature above 75-78°C leads to the degradation of pectins [21], which results in the deterioration of their physical and chemical properties and a significant reduction of their yield. If the hydrolysis-extraction lasts longer than 120 minutes, the pectin yield does not increase.

The obtained hydrolyzate was centrifuged for 10 minutes at 3000 rpm to separate the solids and the pectin liquor. The solids were rinsed multiple times with water until their pH was at 7.0, then dried and sifted to obtain insoluble food fibers. The pectin liquor was cooled down to room temperature.

To separate the related ballast substances and improve the extraction, pectins were coagulated with ethanol at a temperature of 8-10°C for 10 minutes. We mixed equal shares of the pectin liquor and a 96.0% solution of ethanol. Under these conditions, the dense residual matter was formed which was then filtered out of the ethanol solution. The next stage, two-phase refining of pectins, was carried out using a 70.0% ethanol solution for 30 minutes, then a 96.0% ethanol solution for 30 minutes.

The pectin was spread thinly on a pan and dried at 18-20°C for 5-6 hours until its moisture content was not more than 13.0%. This temperature was selected because it helps preserve the jellying properties of pectin, as well as its structure, and improves its solubility.

After the drying was completed, pectin was ground to particles of no more than 0.5 mm in size and sifted through No. 140 silk cloth sieve to obtain a light-yellow powder that was packed in paper bags. The pectin was stored at a temperature of 5-20°C and relative humidity of up to 70.0% for up to 12 months.

| Table 1. The yield of pectins extracted from pea grain hulls |
|---------------------------------|
| Factor name | Pea variety |   |
|              | Temp        | Amior |
| Содержание пектин в оболочках, % of dead weight | 3.40±0.03 | 3.83±0.02 |
| Pure pectin yield, % of the hull weight | 2.94±0.10 | 3.37±0.12 |
| Pectin yield (% of maximum possible) | 86.5 | 88.0 |

The pectins extracted from Temp and Amior pea grain hulls had the organoleptic parameters complying with the GOST 29186-91 standard for food pectin. The analysis of the physical and chemical quality properties for the pectins extracted from pea grain hulls showed that they have a high
moisture content (12.29-12.46%) due to the increased hygroscopicity. The main process parameters of pea grain hull pectins are presented in Table 2. The pectins in question are highly etherified and pure and have good gelling capacities.

Table 2. The properties of pectins extracted from pea grain hulls

| Property                        | Pectin from grain hulls | Pea variety |
|--------------------------------|-------------------------|-------------|
|                                | Temp                    | Amior       |
| Pure pectin, %                 | 77.37±0.10              | 78.00±0.12  |
| Ballast substances, %          | 16.04±0.04              | 15.31±0.02  |
| pH of 1% (water) solution      | 3.40±0.01               | 3.50±0.01   |
| Etherification, %              | 61.81±0.21              | 63.03±0.18  |
| Free carboxyl groups, %        | 7.20±0.01               | 7.02±0.01   |
| Etherified groups, %           | 11.66±0.02              | 11.97±0.04  |
| Acetyl groups, %               | 0.38±0.001              | 0.41±0.001  |
| Methoxylated carboxyl groups, %| 11.28±0.02              | 11.56±0.01  |
| Carboxyl groups, %             | 18.48±0.02              | 18.58±0.04  |
| Gelling capacity, °TB          | 234.50±2.20             | 236.00±2.50 |

The analysis of the sorption capacity of the pectin substances from the Temp and Amior pea grain hulls showed that they mostly bind the ions of lead, cadmium, and copper (Figure 2).

Figure 2. Sorption capacity of 1.0% water solutions of pectins extracted from pea grain hulls.

6. Conclusion
The pectin extraction conditions were selected taking into account the physical and morphological properties of pea grain hulls. The yield of pectin obtained using the developed technology is 86.5-88.0% of the maximum possible.

The quality of the pea grain hull pectins complies with the requirements for industrial pectins. They also have a high sorption capacity for heavy metal ions.

The data obtained allow us to recommend pea grain hulls as the raw material for the production of high-quality pectins. We also recommend using these pectins as gelling components in the production of functional foods.
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