study on the extraction process of pectin from grapefruit peel based on steam explosion technology

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abstract. steam explosion technology is used to pre-treat grapefruit peel, and compare it to the traditional acid-ethanol precipitation method. in this paper, through a single factor experiment, four factors are explored: the material-to-liquid ratio, the pH of the extract, the steam explosion pressure, and the steam explosion pressure maintenance time affect the yield of pectin. finally, the best process parameters for extracting pectin are determined: material-to-liquid ratio 1:15 g/mL, the pH of the extract is 2, the steam explosion pressure is 0.8Mpa, and the steam explosion pressure maintenance time is the 90s. at this time, the yield of pectin is 17.50%.

1. introduction

Grapefruit, also known as Wenden, Xiang Luan, and Nei Zi, has been cultivated in China for more than 3000 years as a plant of the family Rutaceae and genus Citrus [1]. It is speculated that the original center of grapefruit is most likely in Southeast Asia, northern India, or the southern part of China. China's grapefruit harvesting area ranks among the world's top [2] and the domestic grapefruit production areas are mainly located in tropical and subtropical regions such as Fujian, Guangxi, and Guangdong. In 2019, China's grapefruit production was about 4.8 million tons, of which grapefruit peel was between 1.6 and 2.4 million tons, and most of the current grapefruit peel is directly discarded, causing a waste of resources and also creating a series of environmental problems. Grapefruit peel is rich in natural pigments, essential oils, dietary fiber, pectin, naringin, and other active ingredients [3]. Numerous in vivo and in vitro studies have shown that these bioactive components contained in grapefruit peel have antioxidant, antibacterial, anticancer, hyperlipidemia, and hypoglycemic effects [4]. The content of pectin in grapefruit peel is relatively rich and can reach 20%-30% of the dry weight of grapefruit peel [5]. The annual trade volume of pectin in the international market is about 30 000 t. According to statistics, China consumes about 5 000 t annually, of which 80% depend on imports [6]. Therefore, the resource utilization of grapefruit peel can not only solve the environmental problems caused by the random disposal of grapefruit peel but also alleviate the current industry status of pectin in China, which mainly relies on imports.

pectin is a polysaccharide with a unique structure, which exists in the cell walls of certain fruit and vegetables. Pectin is often used as a thickening agent, gelling agent, stabilizer and widely used in medicine, food, textile, and daily chemical fields. The wide application of pectin in various fields also makes its demand increasing. According to statistics, the global demand for pectin is growing at a rate of about 15% per year. The traditional raw materials of pectin are mainly derived from citrus, apples, and sugar beets [7]. The traditional pectin extraction method is mainly the acid extraction method, which is based on the principle that under acidic reaction conditions, insoluble pectin can be converted into
water-soluble pectin, thus allowing pectin extraction. However, the acid extraction method also has the disadvantages of long extraction time, high energy consumption, and high extraction cost, which makes it difficult to be fully promoted. In recent years, many researchers have proposed new methods for pectin extraction, such as alkali extraction, ion exchange, enzyme extraction, and ultrasonic extraction. However, these new extraction methods also have certain drawbacks, such as high industrialization cost and ease of environmental pollution due to the influence of equipment [8]. A single pectin extraction method has obvious disadvantages, and modern research has shown that the joint use of multiple extraction methods has become the mainstream of modern pectin extraction methods, such as microwave-assisted extraction and high-pressure treatment extraction [9]. In recent years, steam blasting technology has been widely used in fiber degumming and pretreatment of biomass feedstocks. Steam explosion technology fills the raw material with water vapor at high temperature and pressure, and then suddenly releases the pressure to enable the decomposition of the raw material.

Steam explosion technology with its efficient, green, clean, and other advantages is increasingly used in the pretreatment of food materials. It has been found that steam explosion can break the bonds in the cellulose macromolecule and break the cell wall. It can be seen that steam explosion can maximize the release of pectin from grapefruit peel, but steam explosion technology has not yet been applied to the extraction of pectin from grapefruit peel. This project intends to make the cell wall of grapefruit peel completely broken by steam explosion, and then extract pectin by acid-alcohol precipitation method, to realize the reuse of grapefruit peel resources to provide a theoretical basis.

2. Materials and Methods

2.1. Materials and reagents
The grapefruit peel was sourced from Ma Jia pomelo purchased online, peeled, and air-dried. The air-dried grapefruit peel was placed in an oven and dried to constant weight before use. Anhydrous ethanol and hydrochloric acid were analytically pure.

2.1.1. Instrument and Equipment
QBS-80B SFE testbed, Constant temperature water bath pot, Rotary evaporator, High-speed centrifuge, precision electronic balance.

2.2. Extract pectin from grapefruit peel
Pectin extracted by acid [10-11]. Extraction process: Production → Pretreatment → Hydrolysis → Extract → Concentrate → Precipitate → Centrifuge → Dry → Finished Product

2.2.1. Pretreatment of raw materials
Dried grapefruit peel was accurately weighed to constant weight with a precision electronic balance, cut into small pieces, and placed in a 500 ml conical flask, and an appropriate amount of distilled water was added to the conical flask, which was held at 95°C for 10 minutes to inactivate pectinase. After filtering with nylon gauze, rinse with warm water at about 50°C until the water is colorless and the peel has no odor.

2.2.2. Acid extraction
A total of 3 portions of pre-treated grapefruit peel were placed in a 500 ml conical flask, and then 150 mL of hydrochloric acid solution with pH = 2 was added, and the conical flask was placed in a constant temperature water bath at 95°C for 60 minutes of extraction. The treated sample was filtered by vacuum extraction to obtain the pectin extract, and the collected extract was concentrated in a rotary evaporator, the temperature of which was set to 90°C. The extract was concentrated to one-third of the original volume to obtain the concentrated solution. Then add three times the volume of 95% ethanol solution in the concentrated solution, the ethanol solution can be seen in the process of adding flocculent material precipitation, after standing for 24 hours, put the mixture in a high-speed centrifuge, centrifuged at
4000g for 10 minutes, then discard the supernatant and collect the pectin precipitate, put the obtained wet pectin in an oven at 65°C, dried to constant weight to make dry pectin. The extraction rate of pectin was calculated using the following equation:

\[ p/\% = \frac{M1}{M2} \times 100 \]

Where: \( p \) is the pectin extraction rate, \%; \( M1 \) denotes the mass of dry pectin, g; \( M2 \) denotes the mass of dry grapefruit peel or dry weight of steam-exploded grapefruit peel puree, g.

2.3. Single-factor test

2.3.1. Effect of liquid/material ratio on the pectin extraction yield

extraction, concentration, precipitation, centrifugation, and drying, the yield of pectin was calculated to investigate the effect of steam blasting viz. pressure time on the yield of pectin.

2.3.2. Effect of pH value of extraction solution on the extraction yield of pectin

The pectin was extracted from 10 g of grapefruit peel puree under the condition of steam burst pressure of 1.2 Mpa and steam burst dimensional pressure time of 90 s. Then the puree was placed in a conical flask and 150 ml of hydrochloric acid with pH 1.0, 2.0, 3.0, and 4.0 were added, the extraction temperature was 95 ℃ and the extraction time was 60 minutes. After extraction, concentration, precipitation, centrifugation, and drying, the yield of pectin was calculated to investigate the effect of the material-liquid ratio on the extraction rate of pectin.

2.3.3. The effect of steam explosion pressure on the yield of pectin

The air-dried grapefruit peel, soaked in distilled water, was put into the material bin of the steam explosion test bench, and then the inlet was closed. Saturated water steam was passed in and maintained at pressures of 0.8, 1.2, and 1.6 MPa for 120 s. Then the high temperature and high-pressure valve were opened to release the pressure instantaneously, and the grapefruit peel puree after steam bursting was collected, weighed 10 g and its dry weight was calculated. Then the grapefruit peel puree was placed in a conical flask and 150 mL of hydrochloric acid solution with pH 2 was added, the extraction temperature was 95 ℃, and the extraction time was 60 min. After extraction, concentration, precipitation, centrifugation, and drying, the yield of pectin was calculated, and the effect of steam burst pressure on the yield of pectin was investigated.

2.3.4. The effect of steam explosion pressure maintenance time on the yield of pectin

The air-dried grapefruit peel, soaked in distilled water, was placed in the material bin of the steam blasting test bench, and then the inlet was closed. Saturated water steam was introduced and steam burst was maintained at a pressure of 1.2 MPa for 60, 90, and 120 s, respectively, and then the puree grapefruit peel after the steam burst was collected, weighed 10 g and its dry weight was calculated, and then the pureed grapefruit peel was placed in a conical flask and 150 mL of hydrochloric acid solution with pH 2 was added, the extraction temperature was 95 ℃, and the extraction time was 60 min. After extraction, concentration, precipitation, centrifugation, and drying, the yield of pectin was calculated to investigate the effect of steam blasting viz. pressure time on the yield of pectin.

2.4. Data Processing

All experiments were done with three replications and all data were expressed as mean ± standard deviation, and the data were analyzed using SPSS 26 and plotted using Origin 2019 software.
3. Results & Discussion

3.1. Results of the single-factor test

3.1.1. Effect of material/liquid ratio on the pectin extraction yield

The pectin extraction rates were determined under different feed-to-liquid ratio conditions, and the results obtained are shown in Figure 1. From the figure, it can be seen that the yield of pectin increased and then decreased with the increase of the amount of extraction solution. The low extraction rate of pectin at a feed-liquid ratio of 1:10 g/mL may be due to the low amount of extraction solution, the high viscosity of the raw material, and the slow molecular diffusion rate [12], which makes it difficult to transfer pectin from the raw material to the extraction solution. In the range of 1:10 to 1:15 g/mL, the extraction rate of pectin increased with the increase of the ratio, and the extraction rate of pectin reached the maximum value when the ratio was 1:15 g/mL, and the maximum extraction rate was 17.50%, which may be due to the increase of the amount of the extraction solution to produce a greater concentration gradient between the raw material and the extraction solution, and then the diffusion rate of pectin was accelerated[13], which increased the extraction rate. This is because the concentration of pectin dissolved is too low due to the large ratio of the feed to liquid, which leads to a longer concentration time, and the pectin components are destroyed during the concentration process, resulting in a lower extraction rate [14]. Therefore, 1:15 was chosen as the best ratio of material-liquid ratio.

![Figure 1. Effect of feed-to-liquid ratio on the extraction rate of pectin](image)

3.1.2. Effect of pH of extraction solution on the extraction rate of pectin

The extraction rate of pectin was measured under different pH conditions of the extract solution, and the results shown in Figure 2 were obtained. From the figure, it can be seen that the extraction rate of pectin reached the maximum value of 17.50% at pH 2. Thereafter, the extraction rate of pectin gradually decreased as the pH of the extraction solution increased. The reason for this change may be that when the extraction solution reaches a certain acidity, it can make the pectin in the raw material transferred to the extraction solution. Too high pH of the extraction solution may affect the conversion rate of pectin into water-soluble pectin to reduce its extraction rate. And too low pH of the extraction solution may lead to the depolymerization of pectin under strongly acidic environmental conditions, resulting in a decrease in its extraction rate.
3.1.3. Effect of steam explosion pressure on the extraction rate of pectin

The extraction rate of pectin at different steam blasting pressures was measured and the results shown in Figure 3 were obtained. It can be seen from the figure that the extraction rate of pectin was significantly increased after steam blasting, and the extraction rate of pectin reached a maximum of 16.88% at a steam blasting pressure of 0.8 MPa, while the pectin yield obtained by the conventional acid extraction method was 10.21%, and the extraction rate of pectin increased by 65.33% after steam blasting pretreatment. The reason may be that steam blasting depolymerized the fibrous structure in the cell wall of grapefruit peel, and its polymerization degree then decreased, making the extraction solution in full contact with the raw material, so that more pectin was dissolved and thus the extraction rate of pectin increased. In the range of steam explosion pressure of 0.8~1.6MPa, the extraction rate of pectin decreased significantly with the increase of steam explosion pressure, showing a decreasing trend step by step, probably due to the excessive steam burst pressure to degrade the pectin into an oligosaccharide, which could not be precipitated and precipitated in the ethanol solution, so that the extraction rate of pectin decreased.

3.1.4. The effect of steam explosion pressure maintenance time on the yield of pectin

The pectin extraction rate was measured at different steam blasting maintenance times, and the results shown in Figure 4 were obtained, from which it can be seen that the extraction rate of pectin did not change significantly at the 60s and 90s, and reached a maximum of 17.30% at 90s of steam blasting maintenance time. The extraction rate of pectin decreased significantly with the extension of steam blasting V.P. The reason may be that the steam blasting time led to the occurrence of caramelization reaction of pectin and thus reduced the extraction rate of pectin.
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Figure 4. The effect of steam explosion pressure maintenance time on the yield of pectin

4. Conclusions
A preliminary investigation of the process conditions affecting the pectin extraction rate was carried out by pretreating grapefruit peel with steam blasting technology in combination with the traditional acid-ethanol precipitation method. Through the single-factor experimental study, the optimal parameters for the pectin extraction process of grapefruit peel were: the material-liquid ratio of 1:15 g/mL, the pH value of the extraction solution was 2, the steam burst pressure was 0.8 MPa, and the steam burst viz. pressure time was 90 s. The extraction rate of pectin was 17.50%, which was significantly higher than that of the acid-ethanol precipitation method. pectin extraction rate. Therefore, the pectin extraction rate of grapefruit peel will be significantly improved by steam blasting technology with appropriate process parameters. The use of steam explosion technology in pectin extraction has obvious advantages and provides a new idea for the resource utilization of grapefruit peels.

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