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Optimization of acid-assisted extraction of pectin from banana (Musa Acuminata) peels by central composite design

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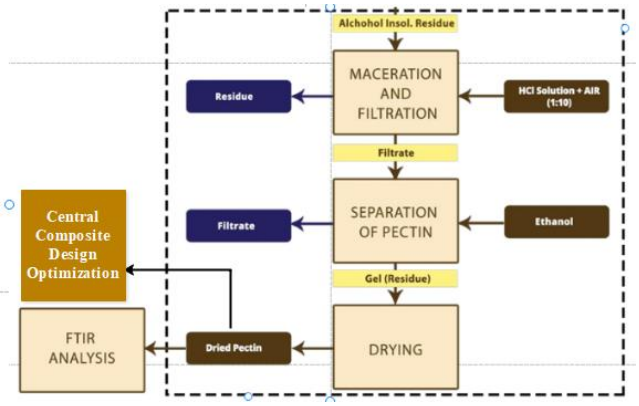
Graphical abstract



Abstract

Pectin extraction from various fruits and its application in food products have been debatable issues for ages. We conducted our research on the biomass wastes of the food industry, primarily banana (Musa Acuminata) peels, in order to develop a modified procedure for the effective extraction of this substance. Banana peels were used to extract pectin using the alcohol precipitation technique. This investigation's goal is to contrast yields of pectin from ripe banana peels by varying the conditions of temperature, time, and pH. Pectin was extracted from banana peels with hydrochloric acid and a central composite design was used to determine the effects of pH (1.5–2.5), extraction temperature (40–85°C), and time (90–240 min) on the yield and purity. The provenience procedure was improved by the removal of phenols, flavonoids, fats, oils, wax, and pigments well before the extraction of pectin. It results in better purity and high yield of Pectin. Soxhlet extraction was initially used to remove them followed by maceration of Alcohol Insoluble Residue (AIR). An FTIR spectrum of the extracted pectin was compared with the commercial pectin and results are reported. Different techniques and methodology are also discussed in this study.

Keywords: Central composite design, pectin extraction, pH, yield, temperature, time, hydrochloric acid, optimization, Design Expert 12.0, Soxhlet apparatus, FTIR spectra.

1. Introduction

Banana is one of the world's most important crops that grow in a range of environments and produces fruit all year round (Maduwanthi and Marapana, 2019; Padam *et al*., 2014). Pakistan is a key player in the banana industry, with more than 349,000 hectares of cultivation. 90% of this land lies in the Sindh province in the southeast of the country. The average area under the fruit in Pakistan was 33,200 hectares with a production of 1,59,400 tons during 2012-2013 (Irfana Noor *et al*., 2015). Average sized banana of approximately 180 grams contains 64 grams of peel and 116 grams of flesh. So, around 55,790 tons of peels are produced in Pakistan according to a 2012-2013 study, and go nowhere but into waste (Hikal *et al*., 2022). Generally, bananas are consumed raw. Their processed form like banana flour, chips, and puree is employed to produce a variety of other products like; smoothies and nectars. The peels of the banana constitute about 30% of the fruit (Sagar *et al*., 2018). Due to the high percentage of nitrogen and phosphorous in bananas, they represent a serious environmental problem (Nonga *et al*., 2011; Sial *et al*., 2019). Additionally, high water content made them susceptible to microbial degradation (Gumisiriza *et al*., 2017). Interestingly, banana peels can be consumed to produce valuable compounds such as pectin, cellulose, and phenolic compounds (Gumisiriza *et al*., 2017; Pathak *et al*., 2016). Consequently, the use of this kind of biomass is not only important from an economic point of view but also crucial from an environmental perspective.

Extraction of pectin hence is considered to be the most efficient utilization of fruit peel wastes. Pectin is a unique ingredient that is used in jams & jellies, marshmallows, marmalades, and a host of other delicacies gel

(Tsykhanovska *et al*., 2021). It is extracted from the citrus waste residue along with some other value-added products including essential oils, flavonoids, and limonoids, and the production of dried cattle feed pellets (Mamma and Christakopoulos, 2014; Sharma *et al*., 2017). The extraction of orange juice yields 55% juice with 45% wet mass residues left over, resulting in a large amount of waste material for disposal (Pacheco *et al*., 2019). Because of the success found in the citrus industry for the extraction of pectin from citrus fruits peel, it is of interest to explore pectin extraction from banana peels (Pacheco *et al*., 2019; Swamy and Muthukumarappan, 2017; Emaga *et al*., 2008).

Pakistan is a country that produces no pectin and adds millions of dollars for importing pectin for its food industries to run as there is no substitute for that. This study is done in context to optimize the process for acid-enhanced extraction of pectin from banana peels. Generally, the pectin obtained from banana peels is very low but the pre-removal of undesired by-products and drying method resulted in an increased amount of pectin. This process of extraction of pectin used is not only efficient in terms of yield but also in terms of purity (Kamble *et al*., 2017).

Several studies have been carried out to investigate the effects of process variables on pectin extraction from banana peels. The effects of pH, temperature, and extraction time were investigated thoroughly (Qiu *et al*., 2010). Similarly, extraction time, pH, and temperature variables were also evaluated by using sulfuric acid to extract pectin (Emaga *et al*., 2008). However, the use of Hydrochloric Acid (HCl) for extracting pectin from banana peels and its effect on process variables has not been yet reported. In the intended study once, the pectin will be extracted from banana peels it will be analyzed using FTIR spectroscopy.

Figure 1. (a) Banana Peels Sun Drying & (b) Dried Banana Peels

2. Material and methods

2.1. Preparation of banana peel powder

The locally available bananas were purchased, and peels were taken off from the flesh. The peels were then cleaned from any debris or adhesive pulp. To extract the pectin, it is necessary to remove the moisture content from the peels. Hence, after cleaning they were allowed to dry in the sun for 2-3 days until the constant weight was obtained. Figure 1 (a) & (b) show before and after dried peels.

The dried peels were milled in a centrifugal grinding mill and meshed with a mesh size of 450 microns resulting in <450 microns particle size. The dried powder, termed Banana Peel powder was ultimately transferred to air-tight bottles at 6°C until further use (Figures 2 and 3).

Figure 2: (a) Food grinder & **(b)** Banana peel powder

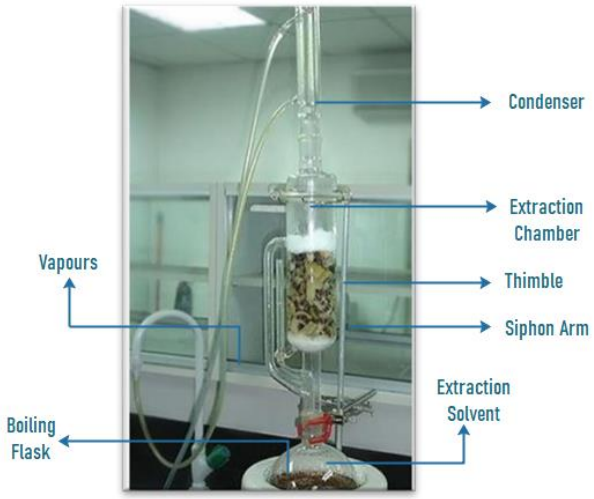


Figure 3: Experimental setup labelled diagram of soxhlet apparatus

2.2. Processing on BPP (banana peel powder) to AIR (alcohol insoluble residue):

2.2.1. Soxhlet extraction

Pectin extraction requires the removal of fats, oils, sugars, carbohydrates, and polyphenols before pectin itself. n-Hexane and ethanol were used for the extraction of oils and sugars respectively from the banana peels powder. The extraction of oils/sugars was carried out until there is no yellow/brown color to be seen in the extractor and capillary tube of the soxhlet apparatus. The extractive-free powder was dried at room temperature and termed Alchohol Insoluble Residue (AIR). It was stored in sealed glass bottles at a temperature of 6°C.

2.3. Rotary vacuum evaporation

The removed component (oils/sugars) collected in the form of solution (n-hexane/ethanol) in the still of the Soxhlet apparatus were sent to the rotary vacuum evaporator to recover the solvent used. This vacuum separation results in the purity of the solvent recovered as the low temperature and low external pressure can increase the volatility difference between the components

present. The remains (oils/fats) of this process are then collected and heated at 40°C to constant weight. Banana oil can be used as a secondary product of our research (Figure 4).

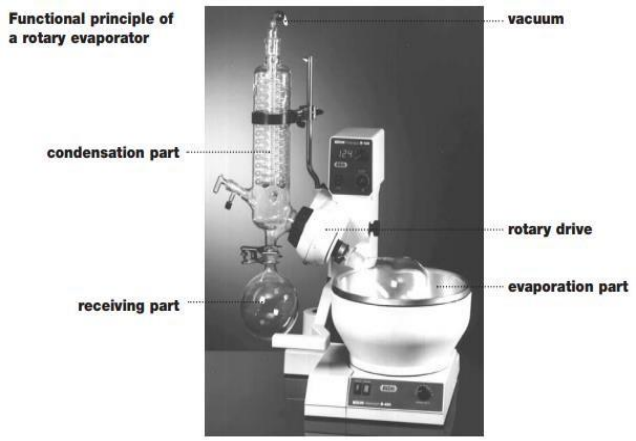


Figure 4: Rotary evaporator

2.4. Extraction of pectin

2.4.1. Maceration

The pectin was extracted from the ALR with the hydrochloric acid solution according to a central composite design (CCD) with 3 variables: pH of the hydrochloric acid solution, time of extraction, and temperature. Temperature ranges from 40°C to 85°C, pH from 1.5 to 3.5, and time from 40 min to 240 min was taken to make 20 experiments. For each extraction, 3g AIR was taken and added to the HCl solution and put on a hot stirrer plate at a stirring rate of 500 rpm along with a thermocouple attached for continuous monitoring of the temperature of the solution, at the required temperature and time of extraction (Figure 5).

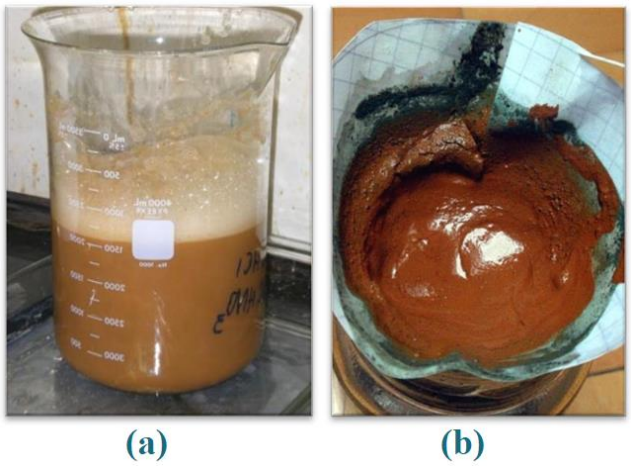


Figure 5. (a) Maceration of air in acid solution (b) Filtration process after maceration

After maceration, the hot solution was filtered by washing with the same HCl solution 2-3 times and the filtrate (Pectin-HCl-Water Solution) obtained was allowed to cool. Absolute Ethanol was added to the filtrate to precipitate out the pectin in the form of a gel. The ethanol to filtrate ratio used was 2:1. The solution formed was stored at 4°C in the refrigerator for 24 hrs and filtered. The gel formed was filtered and then dried. The yield was noted based on AIR.

$$\%Yield = \frac{Weight\ of\ Dried\ Pectin\ (milligram, mg)}{Weight\ of\ AIR(milligram, mg)}$$

The yield obtained from each was compared and after plot fitting, the model of the system was generated using a Central composite design.

3. Results and discussion

3.1. Analysis of pectin

3.1.1. FTIR spectroscopy

FTIR (Fourier Transform Infrared Spectroscope) is the final step used for the analysis. Spectrographs of banana peel powder, alcohol insoluble residue, and pectin show the major peaks of the functional groups obtained. The pectin spectrograph was compared with the reported commercial pectin in the literature to see changes in spectra during the extraction process. The ash content of the pectin was also determined by taking 1g of pectin in the crucible and heated in the furnace at 600°C. The residue obtained was cooled in the desiccator and weighed till the constant weight is achieved. For moisture content, 1g of pectin was weighed and dried at 100°C for 4 hrs. to constant weight (Figures 6 and 7).

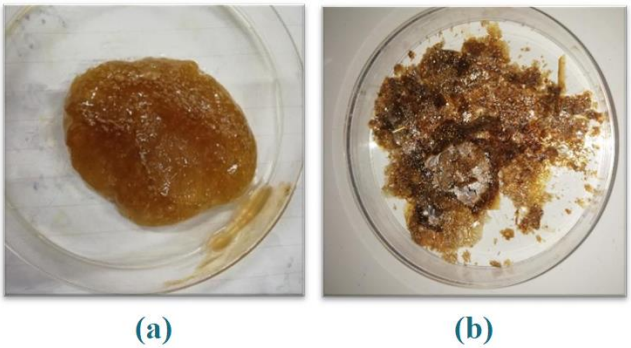


Figure 6. (a) Banana pectin gel (b) Dry banana pectin

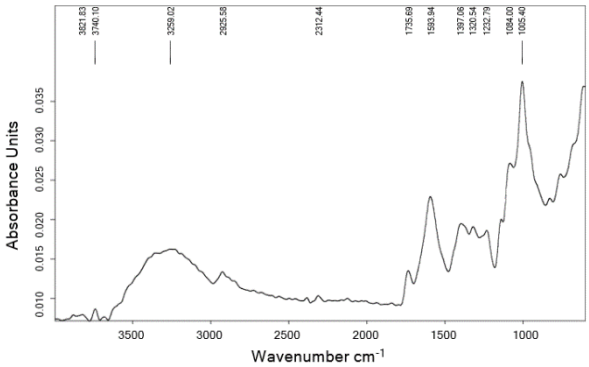


Figure 7. FTIR Spectral Results for Banana Pectin

3.1.2. FTIR spectral interpretation

The characteristic bands around 3000–3500 cm^{-1} denote O-H stretching. The broad absorption observed in our samples compared to the commercial pectin may be due to hydrogen bonding of hydroxyl groups and carboxylic

acid dimers of polysaccharides. The band at around 2925 cm^{-1} is due to the C-H stretching of the galacturonic ring. Bands around another important region that helps in the detection of esterified vs esterified (methylated) functional groups is the fingerprint region 1735−1320 cm^{-1} (Figure 8).

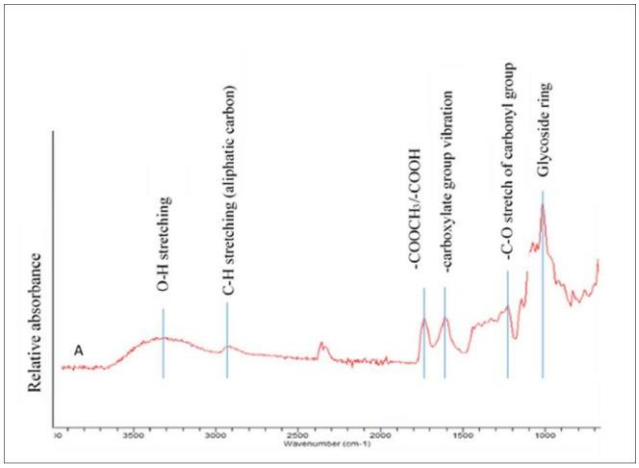


Figure 8: FTIR Spectra of Commercial Pectin

Both the commercial sample and the extracted pectin produced an intense band around 1735 cm^{-1}. The patterns were found to be well correlated to that of commercial pectin (Banerjee *et al*., 2016). The change in intensity of the predicted −COOCH3/−COOH region may be related to the lower degree of esterification in the case of maceration of banana extracted pectin. The band around 1593 cm^{-1} is due to the carboxylate group. Small bands at around 1200 and 1084 cm^{-1} may be due to the glycoside ring, C-O stretching, and OH bending. Bands around 650 cm^{-1} may be due to the presence of the pyranoid ring structure of pectin.

3.2. Optimization of extraction process using central composite design methodology

Optimization of the extraction process for pectin revealed that hydrochloric acid at low pH is more efficient to extract pectin. This is due to the ionic strength of HCl which is higher than weak acids such as citric. Given the same concentration, higher ionic strength acids have more capability to precipitate pectin due to their higher affinity for cations such as Ca^{2+} which stabilizes the pectin molecule. Thus, given enough time, a higher amount of pectin can be precipitated out (Emaga *et al*., 2012).

The moisture content of the banana peel pectin did not vary significantly from the commercial citrus pectin. Low moisture content in pectin is necessary for safe storage as well as to inhibit the growth of microorganisms that can affect the quality due to the production of pectinase enzymes.

Table 1. Central Composite Design Methodology In Design Expert software for extracted Pectin in different time, temperature and pH for average yield

Run	pH	Temperature	Time	Yield (%) For RUN 1	RUN 2	Average
1	2.5	62.5	165	10.37	9.73	10.05
2	3.5	62.5	165	9.53	10.84	10.185
3	2.5	62.5	165	8.453333	9.17	8.811667
4	2.5	62.5	90	8.626667	10.73667	9.681667
5	2.5	62.5	165	9.736667	8.696667	9.216667
6	2.5	40	165	6.763333	8.026667	7.395
7	1.5	40	90	5.723333	4.056667	4.89
8	1.5	62.5	165	11.09	11.21667	11.15333
9	1.5	85	90	11.74	12.26333	12.00167
10	2.5	62.5	165	10.83333	9.186667	10.01
11	3.5	85	90	10.67	16.16667	13.41833
12	3.5	40	240	9.423333	8.776667	9.1
13	2.5	62.5	165	10.77333	12.7	11.73667
14	2.5	85	165	10.69667	10.25333	10.475
15	2.5	62.5	165	8.733333	8.193333	8.463333
16	1.5	85	240	15.17333	8.656667	11.915
17	3.5	40	90	9.4	8.66	9.03
18	1.5	40	240	6.783333	6.626667	6.705
19	2.5	62.5	240	10.24333	8.986667	9.615
20	3.5	85	240	10.47333	12.14	11.30667

Lower ash content was also obtained from peels which implies that it can form better gels, the maximum limit though, for the good quality gel is 10% (Ismail *et al*., 2012). Commercial citrus pectin has a very low ash content (1.76%) compared with banana peel pectin Thus, the gel quality that will be produced from these pectins would vary, with banana peel pectin expected to be of lower quality (Girma, 2017). This could, however, be improved through the extraction procedure, if a more efficient acid extraction will be employed to chelate more Ca^{2+} which contributes to the majority of the ash content. The Table 1 presents the responses for all the experimental runs.

The extraction yield values ranged from 6 to 15% (w/w, based on the dry weight of (AIR), within the range

reported, which was from 2.4 to 21.7 wt% by [20]. The yield was increased by increasing temperatures and lowering pH while the time did not have a significant impact as compared to that reported earlier (Chan and Choo, 2013). Other studies, (Aina *et al*., 2012) also demonstrated that harsh conditions favored pectin extraction yield from apple pomace, lemon by-product, and durian rind, respectively. A very low pH (1.5) was demonstrated to result in higher extraction of low molecular weight compounds when compared to pH 2.0, i.e., the purity of extracts was impaired by too acidic conditions (Anhwange *et al*., 2009).

4. Conclusion

Pectin was successfully extracted from banana peels with hydrochloric acid, under different conditions of pH, temperature, and extraction time. Harsh temperature and pH conditions resulted in higher extraction yield. The optimum conditions of pectin extraction which produced a maximum yield of pectin were: 85°C, 240 min, and pH 1.5.

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