

Extraction of Pectin from Orange Peel

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ABSTRACT

Agricultural by-product like orange peel play an important role in pectin manufacture. Pectin was extracted from sweet orange peel using citric acid at two different temperatures 70 and 80 °C, pH 1.0 and 2.0 and extraction times 10 and 30 min, using a 2² factorial design. Temperature, pH and extraction time had highly significant effect on the pectin yield. Model fitting and analysis to optimize the extraction process conditions. Pectin yields varied from 49.00% to 54.15%. The optimal conditions for maximization of pectin yield at 80 °C and pH 1.0 with an extraction time of 10 min considering model extrapolation.

1. Introduction

Pectin is used as a natural gelling agent and is a versatile solution for addressing various health issues. It is commonly employed in the food and pharmaceutical processing industries and serves as a thickening agent in a variety of food products. Pectin contributes to the texture and taste of healthy food production. Agricultural by-products, such as orange peel, are integral to the manufacturing of pectin. Oranges are grown widely around the world, and the cultivation of orange trees occurs in both tropical and subtropical climates. Additionally, oranges are processed for juice extraction and fragrant peel. Despite its widespread industrial use, there is no local production of pectin in Nigeria, highlighting the need for an investigation into the local production of pectin from orange peel.

The project focused on extracting pectin from orange peels. The extraction process was conducted at different temperatures, pH levels, and extraction times, and the impacts of these factors were analyzed. A review of the literature on pectin and the extraction process was performed. The necessary chemicals and equipment were selected for the experiment. Fresh sweet oranges were used as the raw material for the experiment. The pectin extraction process was carried out using a Soxhlet apparatus.

A full factorial experimental design was implemented at temperatures of 80°C and 75°C, extraction times of 30 minutes and 10 minutes, and pH levels of 2 and 1. Pectin characterization was performed by examining the degree of esterification and galacturonic acid content. Finally, FT-IR analysis was conducted. The optimal yield was obtained at a temperature of 80°C, an extraction time of 10 minutes, and a pH of 1, leading to the conclusion of the parameters.

1.2 Objective

The primary goal of the research was to extract pectin from orange peels under different conditions. Additional objectives included:

- Optimizing pectin yield by maintaining the optimal experimental conditions.
- Enhancing the application of agricultural by-products, such as orange peels, in pectin extraction.

1.3 Work Nature

I conducted a comprehensive investigation into pectin, encompassing its extraction techniques and properties. I gained an understanding of the chemical structures of various forms of pectin, including high methoxyl, low methoxyl, and amidated pectin. I explored pectin's nutritional value and its applications in

the food processing industry. I reviewed different extraction techniques, focusing on variables that impact the extraction rate, such as temperature, pH, and solvent selection. In the experimental phase, I personally carried out the extraction of pectin from the albedo portion of sweet oranges, adhering to established procedures. I systematically examined the influence of varying parameters—including pH, temperature, and extraction time—on the pectin extraction rates using a factorial design approach. I also took responsibility for analyzing the galacturonic acid content and the degree of esterification of the pectin. Additionally, I used FT-IR spectroscopy for further research.

Organization Chart

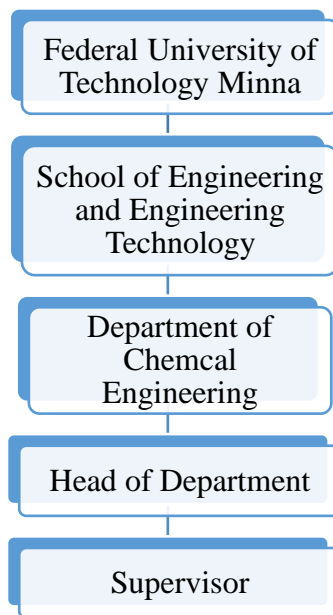


Figure 1 Organization Chart

1.4 Duties

- To investigate the impact of temperature extraction time and pH for the extraction of pectin.
- To develop the model of linear regression to create the relationship between the pectin.
- To characterize extraction of pectin from orange peels.
- To perform the FTIR analysis of the pectin.
- To prepare the final technical report including all the tasks.

1.5 Personal Engineering Activities

I searched the literature for details regarding pectin extraction methods. I researched the fundamentals of pectin extraction and gathered ideas related to pectin. My understanding of the chemical structure of pectin was enhanced by my studies in chemistry. I explored pectin in its various forms, including amidated, low methoxyl, and high methoxyl. After researching its nutritional value in more detail, I learned that pectin is a nutrient-dense fiber source with anti-cancer, cholesterol-regulating, and mineral-binding qualities. I reviewed a list of foods high in pectin, which included items such as apples, oranges, and apricots. I also discovered that pectin has various applications in the food processing industry. Additionally, I researched pectin extraction techniques, investigating several methods, including continuous and batch processes.

1.6

After that, I began investigating how various factors affected the rate of pectin extraction. I selected the appropriate chemicals, tools, and raw materials. For the experiment, I chose substances such as ethanol,

sulfuric acid, citric acid, n-hexane, and distilled water. I decided to conduct the experiment using the Soxhlet extraction unit and selected additional equipment, including a tray dryer, pH meter, measuring cylinder, pipette, beaker, potentiometer, magnetic stirrer and incubator. I acquired these chemicals and equipment from the WAFT laboratory. Furthermore, I collected fresh sweet oranges from Kure Market in Minna, Nigeria. After extracting the juice at the food processing pilot plant, I processed the obtained peels to separate the albedo portion (rich in pectin) from the flavedo (oil and pigment). I minced the albedo portion and washed it with cold water to remove any adhering juice. Finally, I air-dried the albedo portion and ground it prior to pectin extraction.

2. Methodology

Then, I began the pectin extraction process following standard procedures. I defatted the ground raw material using hexane in a Soxhlet apparatus. I thoroughly mixed the raw material with water at different pH values, adjusting the mixture's desired pH with 0.1N citric acid. Next, I incubated the mixture at various temperatures for different time intervals while stirring frequently. After the incubation process, I collected the supernatant and filtered out the residue using filter cloths. I concentrated the supernatant in a rotary evaporator, then precipitated the obtained concentrate with ethanol (two volumes). Finally, I dried the extracted pectin in a vacuum oven at 40°C to achieve a constant weight and then finely ground the extracted pectin.



Figure 2 Precipitation of pectin pictorial presentation



Figure 3 Obtained dry pectin

3. Results

Following that, I began the experimental design procedure. I employed a two-level full factorial design (2^3) to investigate the impact of variables on pectin extraction. I selected pH, temperature, and extraction time as the variables affecting pectin yield, with two levels for each parameter. The selected temperatures were 80°C and 75°C, extraction times of 30 minutes and 10 minutes, and pH levels of 2 and 1. Next, I chose both commercial low-methoxy pectin (LMP) and pectin samples extracted under optimized conditions for analysis. I determined the degree of esterification of the pectin using FT-IR spectroscopy and evaluated the galacturonic acid content using the colorimetric method. I dissolved the samples in distilled water (0.5 mg per mL) and stirred gently. To prepare 400 mL of the sample, I added 40 mL of a 4.0 M sulfuric acid-potassium sulfate solution (pH 1.6) and mixed them vigorously. I then added H₂SO₄ with 75 mM sodium tetraborate (2.4 mL) and stirred the solution thoroughly. I incubated the solution for 20 minutes in a water bath. After that, I cooled the mixture and added 40 mL of 0.15% (w/v) m-hydroxyphenyl in 0.5% (w/v) NaOH, followed by thorough stirring. I observed the development of a pink color after 5 to 10 minutes and measured the absorbance at 525 nm using a standard curve with galacturonic acid. Finally, I conducted FT-IR spectral studies, recording the FT-IR spectrum of pectin using the Shimadzu 8400S FT-IR spectrometer.

3.1

Lastly, I interpreted the results to examine the impact of process variables on pectin extraction. I obtained the reaction parameters using the Design Expert 8.0.7.1 software. I analyzed the pectin yield from the orange peels under different extraction conditions. First, I assessed the impact of temperature and found that pectin yield increased with rising temperature. I recorded a pectin yield of 50.50% at 70°C and a yield of 54.15% at 80°C. Next, I examined the effect of pH on pectin yield and observed a decrease in yield as pH increased from 1 to 2. I achieved a pectin yield of 54.15% at a pH of 1 and 49.80% at a pH of 2. Furthermore, I found that the reaction time for pectin extraction depended on the temperature and pH of the process. I obtained the results of ANOVA from the Design Expert 8.0.6 software, which indicated that temperature and pH had main effects of 1.06 and 2.51 on pectin yield. The contributions of temperature and pH were 4.62% and 25%, respectively. Based on these outcomes, I concluded that pH had the highest effect on pectin yield. I reviewed the ANOVA results and determined that all three operating parameters significantly increased the yield. I observed that the optimized critical values were 80°C, 10 minutes, and a pH of 1, resulting in a yield (Y_p) of 54.1%. From the FT-IR analysis, I obtained a spectrum with wavelengths ranging from 950 to 1200 cm⁻¹.

Table 1 Yield of Pectin from sweet orange

Run	Temperature	Time	pH	Response
1	75	10	1	50.50
2	80	10	1	54.15
3	75	30	1	50.95
4	80	30	1	50.25
5	75	10	2	51.65
6	80	10	2	49.80
7	75	30	2	50.55
8	80	30	2	49.00

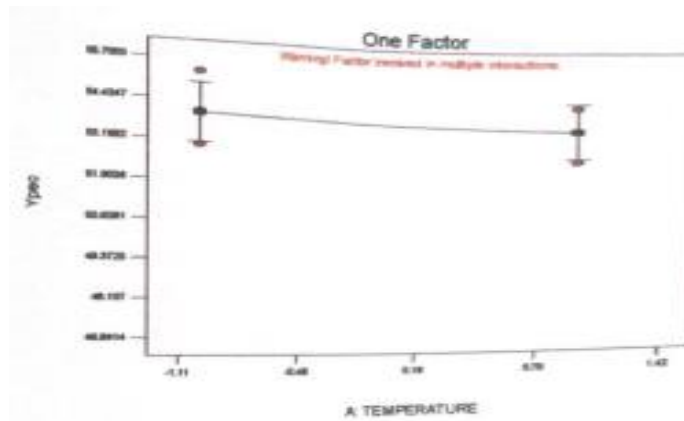


Figure 4 Effect of temperature

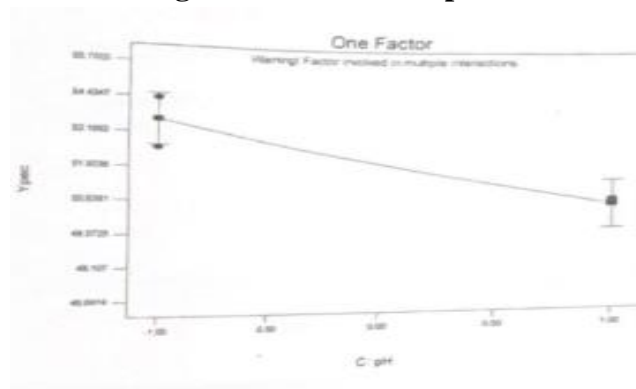


Figure 5 Effect of pH

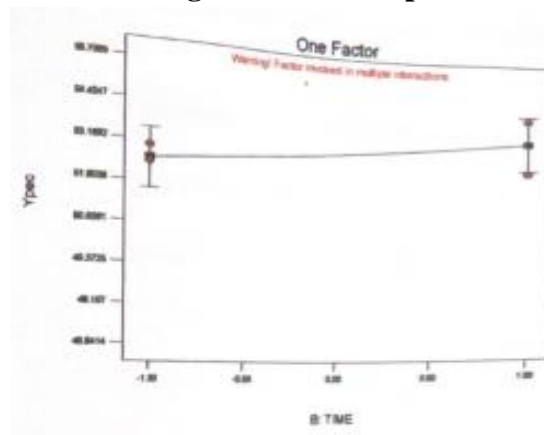


Figure 6 Effect of reaction

Table 2 Regression coefficient and significance of response surface linear

Term	Std	Sum of Square	%Contribution
A	1.06	4.52	4.62
B	2.51	25.25	25.85
C	-3.65	54.39	55.68
AB	-1.24	6.13	6.27
AC	0.21	0.18	0.18
BC	0.26	0.28	0.28
ABC	0.26	0.28	0.28

Table 3 Analysis of variance

Lack of lift		0.000	0.000
Pure error		6.66	6.82

Table 4 Model Summary statistics

Source	Sum of squares	Mean squares	F value	p-value	Df
Model	90.74	15.12	19.61	0.0001	6
A	4.52	4.52	5.86	0.0386	1
B	25.25	25.25	32.74	0.003	1
C	54.39	54.39	70.53	<0.0001	1
AB	6.13	6.13	7.94	0.02015	1
Residual	6.94	0.77			9
Lack of Fit	0.28	0.28	0.33	0.5810not significant	1
Pure Error	6.66	0.83			8
Cor Total	97.68				15

3.2 Problems and Solutions

I encountered a complication during the preparation of the raw material for pectin extraction. I discovered that the raw material had adhering juice, which affected the extraction process. I realized that I could not extract the pectin effectively due to this adhering juice. To identify the flaws in the raw material preparation process, I reviewed the steps involved. Additionally, I researched preparation methods from previous literature to determine the correct approach. I also discussed the issue with my mentor and gained his insights. Following my research, I found that the problem arose after the separation of the albedo and flavedo portions. I learned that the adhering juice could be removed by vigorous washing. Therefore, I minced the albedo portion and washed it thoroughly with cold water. Afterward, I checked the raw material and confirmed that it no longer had any adhering juice.

3.3 Creative Works

After extracting the juice at the food processing pilot facility, I treated the peels to separate the albedo (rich in pectin) from the flavedo (oil and pigment). I cut the albedo portion into small pieces and rinsed it under cold water to remove any adhering juice. To achieve a consistent weight, I dried the extracted pectin in a vacuum oven at 40°C.

3.4 Project Management

I assembled a diverse team by carefully selecting members with expertise in chemistry, food science, laboratory work, and project management. I defined my roles and responsibilities, ensuring that skills matched the project's requirements. I conducted an initial meeting to introduce the project, set its goals, and clarify the expected outcomes. I discussed the project timeline, milestones, and deadlines. I established clear communication channels and selected preferred collaboration tools based on input from my supervisor. I led the development of a comprehensive project plan, outlining the step-by-step process for

pectin extraction. I worked collaboratively to identify potential risks and formulate mitigation strategies. Additionally, I was involved in defining the project budget and resource requirements.

3.5 Codes and Ethics

I reviewed the ISO 11014:2009 code, which helped me handle the various chemicals used in the project. I also consulted the university's code for further guidance.

3.6 Conclusion

A comprehensive investigation into pectin, its extraction techniques, and its properties was conducted. The chemical structures of various forms of pectin, including high methoxyl, low methoxyl, and amidated pectin, were examined. The nutritional value of pectin and its applications in the food processing industry were also explored. Various extraction techniques were reviewed, and factors affecting the extraction rate, such as temperature, pH, and solvent selection, were analyzed. In the experimental phase, pectin was extracted from the albedo portion of sweet oranges following established procedures. The impact of different parameters—including pH, temperature, and extraction time—on pectin extraction rates was investigated using a factorial design. The degree of esterification and galacturonic acid content of pectin were analyzed, and FT-IR spectroscopy was employed for further studies. The results revealed that temperature and pH significantly affected pectin yield, with pH being the most influential factor. The optimized conditions for pectin extraction were identified as 80°C, 10 minutes, and a pH of 1. FT-IR analysis provided valuable spectral data within the range of 950 to 1200 cm^{-1} . Through this project, I developed my skills in management, supervision, direction, communication, and leadership while executing the extraction of pectin from orange peel.

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