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Extraction of Pacitan Sweet Orange Peel Pectin as a Bioadsorbent on the Adsorption Process of Fe Metal in $FeCl_3$ Solution

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ABSTRACT

One alternative to treating waste that contains heavy metals is by bioadsorption process from natural materials as adsorbents. In this study, Pacitan sweet orange peel raw material was used as a $FeCl_3$ metal bioadsorbent. This study aims to obtain pectin as a bioadsorbent for Fe metal in FeCl3 solution with the influence of extraction time and temperature on pectin extraction. In this study, pectin was extracted using the direct extraction method with HCl solvent. To prove the characteristics of pectin, FTIR analysis was conducted by observing the C=O group contained in pectin. Atomic Adsorption Spectrophotometer (AAS) analysis was then used to perform pectin adsorption in a FeCl3 solution. The research results show that optimum conditions are achieved at a temperature of 80°C, a contact time of 90 minutes with the addition of a pectin mass of 90 mg obtained an adsorption efficiency of 60.52%. From the isotherm adsorption analysis, Freundlich isotherm was applied to the adsorption of $FeCl_3$ metal with a determination coefficient (R^2) of 0.968.

INTRODUCTION

Along with industrialization in Indonesia come positive and negative impacts. One negative impact is an increase in environmental pollution due to industrial waste, especially heavy metals that harm water. Heavy metals that pollute the environment and are toxic include chromium (Cr), silver (Ag), cadmium (Cd), lead (Pb), zinc (Zn), mercury (Hg), copper (Cu), iron (Fe), molybdenum (Mo), nickel (Ni), tin (Sn), and cobalt (Co), as well as lighter metals such as arsenic, aluminum (Al), and selenium (Se) (Solika, 2017). Various techniques have been attempted to reduce heavy metal levels, but these methods have drawbacks. Therefore, other alternatives are needed, such as using natural materials as adsorbents through adsorption. One such material is pectin from orange peel waste (Ashraf, 2010).

Pectin is a compound found in plant cell walls, especially in the peels of fruits such as oranges, bananas, apples, and papayas (Murtando, 2016). The albedo section of orange peel contains 16.6% pectin, while the flavedo contains 14.2%. Additionally, the pectin in orange peels is a type of low methoxyl pectin, making it a potential bioadsorbent (Tuhuloula, 2013).

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Pectin, a complex polymer, is found in plant cell walls (Damanik, 2019). It plays a role in intercellular adhesion and maintains the structural integrity of plant cell walls. Plant-derived pectin can be water-soluble, chelator-soluble, or present in the form of protopectin (Alamineh, 2018). Due to its ability to bind to heavy metal ions and form insoluble complexes, pectin is useful as a heavy metal bioadsorbent (Andrini, 2021).

The pectin was separated from the orange peel using an extraction method. Then, FTIR testing was used to analyze the pectin content. FTIR testing identifies chemical bonds in a sample by the various peaks they produce in the spectrum (Vasdazara, 2018). An atomic absorption spectrophotometer (AAS) was used to test the adsorption results to determine the adsorption efficiency of the orange peel pectin sample.

According to Rahayu's (2021) research, pectin extracted by heating at 80 °C for 80 minutes has an adsorption efficiency of 88.096% against the heavy metal Fe with the addition of 50 mg of pectin. This demonstrates that the quantity of metal ions adsorbed increases with the quantity of adsorbent used in the adsorption process. The addition of adsorbent increases the number of active sites, thereby increasing adsorption efficiency; however, the active sites compete with each other for adsorption. Therefore, this study used a variety of adsorbent masses: 50, 60, 70, 80, and 90 mg. Previous research shows that orange peel has a high adsorption efficiency against the heavy metal iron (Fe), so this study aimed to obtain pectin as a bio-adsorbent against iron (Fe) in a $FeCl_3$ solution, considering the effects of time and temperature on pectin extraction. The aims of this study is to obtain pectin as a bioadsorbent against iron (Fe) metal in a ferric chloride ($FeCl_3$) solution, considering the influence of pectin extraction time and temperature.

RESEARCH METHODS

Place and Time of Research

This investigation was carried out from October to December 2024 at the Research Laboratory of the National Development University "Veteran" East Java.

Tools and Materials

The tools utilized in this research are ball coolers, thermometers, three-neck flasks, stirring motors, heating coats, FTIR *(Fourier Transform Infrared),* and AAS *(Atomic Absorption Spectrophotometer).* The materials used in this study are Pacitan Sweet Orange peel, Hydrochloric Acid, Ethanol, Aquadest, and standard $FeCl_3$ Solution.

Pectin Extraction

The orange peel is cut into 2 cm pieces then cleaned and dried in the oven. It is then ground until smooth and weighs 40 grams. The orange peel is put in a three-neck pumpkin and 800 ml of 1N hydrochloric acid is added. Furthermore, extraction is carried out with a stirring speed of 230 rpm with variable temperature (40°C, 50°C, 60°C, 70°C, and 80°C) and time (30 minutes, 60 minutes, 90 minutes, 120 minutes, and 150 minutes). Then the solution is filtered and the obtained filtrate is added ethanol with a ratio of ethanol : filtrate which is 2 : 1 for 24 hours. The deposits that form are filtered and washed using 96% ethanol and then dried in the oven. The pectin results obtained were analyzed by FTIR.

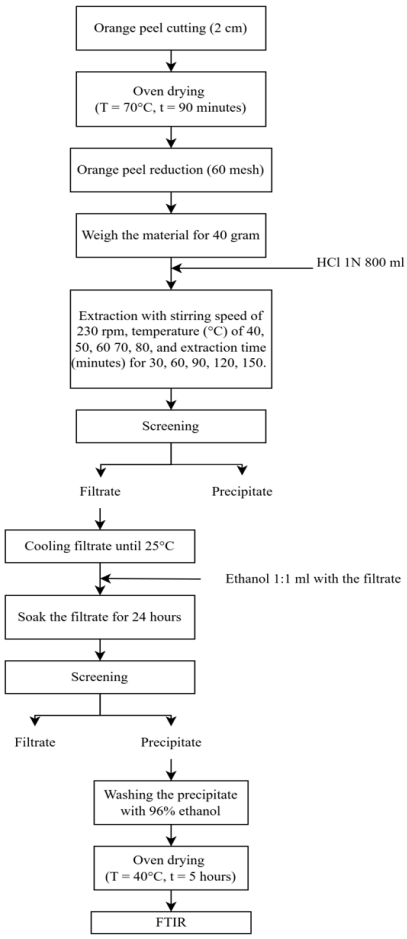

Figure 1. Pectin Extraction Flow Chart

Adsorption Process

Pacitan Sweet Orange Peel Pectin with masses (50 mg, 60 mg, 70 mg, 80 mg, and 90 mg) was contacted with 50 ml of $FeCl_3$ solution 25 mg/L. The mixture was administered at a speed of 230 rpm with a contact time of 120 minutes. Next, the stirred pectin is separated from the Fe solution with filter paper. The filtrate obtained was analyzed with AAS.

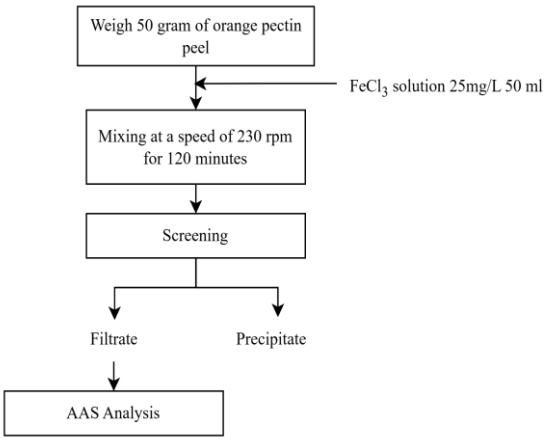

Figure 2. Adsorption Process Flow Chart

RESULTS AND DISCUSSION

Pacitan Sweet Orange Peel Pectin Extraction

Research on pectin extraction of Pacitan Sweet Orange Peel was carried out at the UPN "Veteran" Research Laboratory in East Java. The process of making pectin is carried out in several stages, first the reduction of the size of the orange peel, then extraction with HCl, then the soaking of pectin with ethanol to precipitate pectin, reduce moisture content, purify pectin, and prevent pectin hydrolysis. Then there is the process of washing with ethanol to remove acid residues. The factors tested in this study are the extraction time and the temperature used in the extraction process. The extraction time used is 30 minutes, 60 minutes, 90 minutes, 120 minutes, and 150 minutes with temperatures of 40°C, 50°C, 60°C, 70°C, and 80°C.

The Relationship Between Temperature and Time with Yield

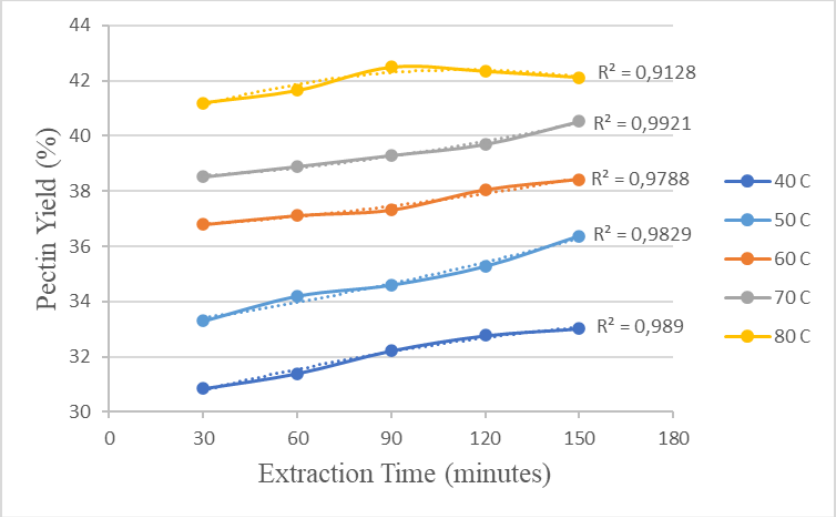

Figure 3. Effect of Temperature and Extraction Time on Pectin Yield (%)

According to the data above, it can be seen that the highest pectin yield is produced at the condition of extraction time of 90 minutes and extraction temperature of 80°C with a yield of 42.5%. Meanwhile, the lowest pectin yield is found in the 30-minute extraction time condition at an extraction temperature of 40°C with a yield of 30.85%. The graph shows that the higher the extraction temperature, the greater the weight of the pectin produced. This is in accordance with the research conducted (Yoakhim, 2021) that higher temperatures will accelerate the process of hydrolysis protopectin from orange peel tissue into water-soluble pectin so that there will be more pectin obtained, however, at an extraction time of 150 minutes and a temperature of 80°C, the weight of the pectin produced begins to decrease. This decrease in pectin yield is caused because hydrogen ions that substitute calcium and magnesium from hydrolyzed protopectin will cause the breakdown of molecules in the galacturonate chain bonds to be released. This is in accordance with the research conducted by (Zahrotun, 2013) where the time of pectin extraction is directly proportional to the amount of protopectin that turns into pectin, but at a certain time pectin can be degraded into pectic acid so the optimum time is important to determine. In addition, too high a temperature causes pectin to not form properly because too high a temperature can damage the structure of pectin molecules and pectin can degrade or decompose so that it loses its ability to form gel.

Results and Discussion of Pectin Functional Clusters with FTIR Analysis

FTIR testing was carried out to identify the degree of esterification of pectin functional groups extracted from orange peel raw materials and extractor solutions in the form of HCl. The range of wave numbers used was 4000-400 $^{cm-1}$ are measured with a prism or grating monochromator (Griffiths, 2007). The FTIR test was carried out on all pectin results that were formed, and the results that were closest to the standard pectin data were selected. In the FTIR test, the results were the closest to standard pectin, namely at a variation of 80°C for 90 minutes. In this analysis, the absorption data of the number of waves in the test pectin was obtained as follows:

Table 1. FTIR Test Results of Research and Standard Pectin

Variable	Pectin Test	Standard Pectin	Information
80°C / 90 minutes	3628,1	3031 – 3793*	OH (Alcohol)
	2929,87	2886,33 – 2973,30**	CH_3 (Aliphatic)
	1735,93	1730 – 1750,41**	C=O (Esther)
	1639,49	1626,49 – 1680**	C=C (Alkena)
	1236,45	1014,40 – 1246,44**	C-O- (Ether)

*Source: (Daud, 2019)
**Source: (Latupeirissa, 2019)

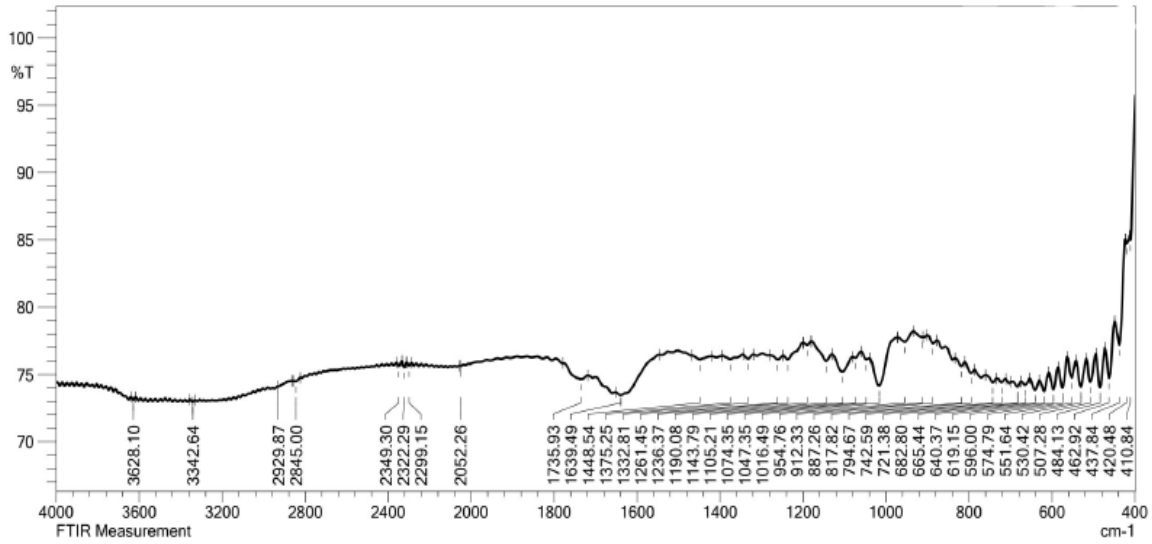

Figure 4. FTIR Test Results on 80°C Sample ; 90 minutes

The results of the FTIR test look like the FTIR spectrum data in table 1, the absorption of the wavenumber in each functional group of the standard pectin and the extracted pectin results are slightly different. In the readable wave, a peak spectrum appears at a wavelength of 3628.1 $^{cm-1}$ which indicates the absorption of the O-H function group or hydrogen bond. Absorption at wave number 2929.87 $^{cm-1}$ indicates the absorption of the -CH_3 group elongation. In the area of wave number 1735.93 $^{cm-1}$, it indicates the absorption of a carboxyl group (-C=O) or ester. There is an absorption at the wave number of 1639.49 $^{cm-1}$ which indicates the presence of absorption from the alkene group (C=O). Meanwhile, there is an absorption from the ether group (C-O-) at a wave number of 1236.45 $^{cm-1}$. Nkafamiya (2019) assert that even minor variations in a molecule's composition and structure can have a major impact on the absorption peaks.

Results and Discussion of Adsorption Against $FeCl_3$ Solution

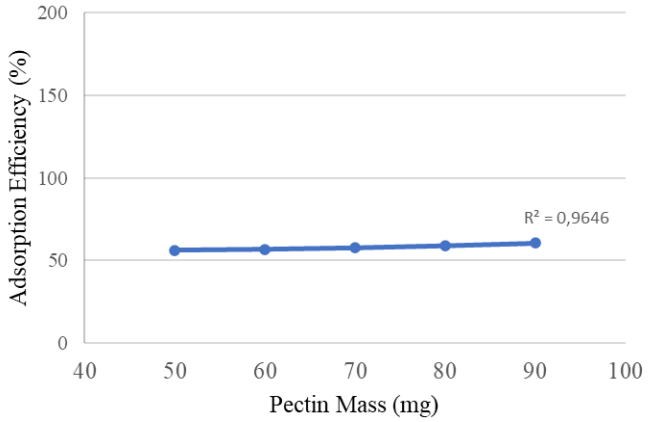

Figure 5. Effect of Pectin Mass (mg) on Adsorption Efficiency (%)

In Figure 5, it can be seen the effect of the addition of pectin mass on the efficiency of adsorption. In the addition of the pectin mass of orange peel as much as 50 mg, 60 mg, 70 mg, 80 mg, and 90 mg, the efficiency values of 56.28%, 56.84%, 57.68%, 59%, and 60.52% were obtained. Based on these data, it can be seen that the more pectin mass is added, the more the adsorption efficiency of $FeCl_3$ metal increases. This is in accordance with the research conducted (Fatmawati, 2018) where the more adsorbents used, the more adsorbent metal ions will also be adsorbed. The addition of adsorbents will increase the active side so that the efficiency of the adsorbent will increase, but the active side competes with each other in the absorption of the adsorbate. In general, the adsorption process occurs on all surfaces, but the absorption efficiency of the adsorption itself is affected by the surface area that is in direct contact with the adsorbate. The magnitude of adsorption is directly proportional to the surface area, the larger the surface area, the more metal ions are adsorbed (Rizki, 2019).

Adsorption isotherms

Adsorption isotherms can demonstrate the connection between the equilibrium concentration of ions in the solution and the quantity of adsorbent ions per weight of the adsorbent. The balance of $FeCl_3$ adsorption by pectin was studied using the adsorption isothermal model of Langmuir and Freundlich. The Langmuir isotherm equation is created by connecting a graph between Ce/qe and Ce. Meanwhile, the Freundlich isotherm equation is made by connecting a graph between Log qe and Log ce. The isotherm model obtained from the results of the study using Pacitan sweet orange peel pectin bioadsorbent is shown in figures 6 and 7.

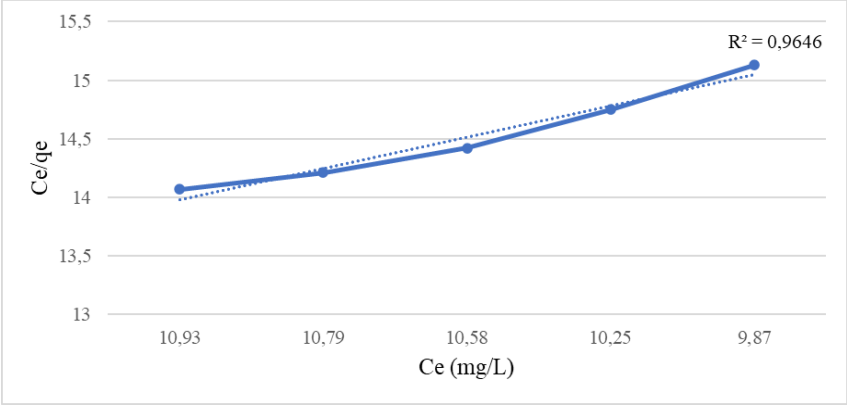

Figure 6. Langmuir Isotherm Adsorption Equation of Ce/qe Versus Ce

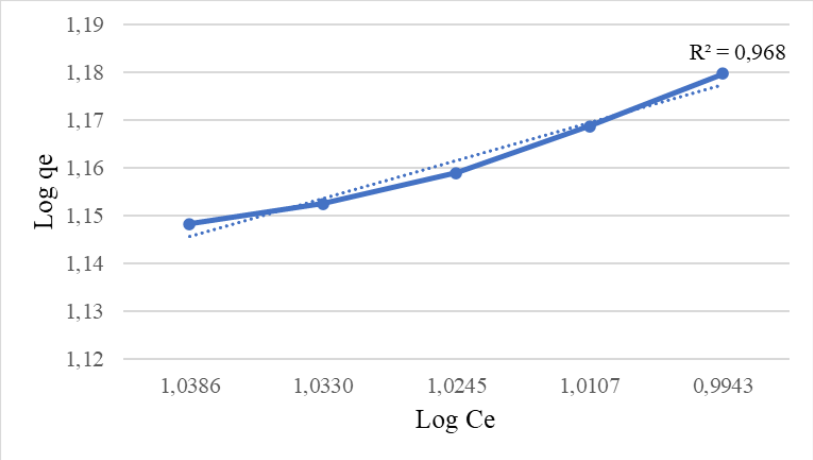

Figure 7. Freundlich Isotherm Adsorption Equation of Log qe versus Log Ce

Testing of the Langmuir and Freundlich adsorption equations is proven by a linearization graph showing the value of the determination coefficient $R^2 \geq 0.9$ (close to 1). In Figure 6, the determination coefficient R^2 for the Langmuir isothermal equation is 0.9646. In addition, the determination coefficient of R^2 for the Freundlich isotherm equation is shown in figure 7 which is 0.968. Based on the determination coefficient value (R^2) obtained from the isotherm images of Langmuir and Freundlich, it is shown that the adsorption that is more in accordance with the adsorption of $FeCl_3$ metal by the pectin of Pacitan sweet orange peel is Freundlich isotherm. This is due to the greater value of the determination coefficient (R^2) of the Freundlich isotherm model, which means that the absorption of metals in the absorption process occurs by fission. Fissorption is an adsorption event that occurs due to the presence of physical forces characterized by small adsorption heat (Apriyanti, 2018). In addition, from figures 6 and 7, the Langmuir equation Ce/qe = 0.266Ce + 13.718 and the Freundlich equation log qe = 0.0079log Ce + 1.1379, so that the constant price of both equations can be calculated.

Table 2. Results of Isothermal Adsorption Calculation

Langmuir Isothermal			**Isoterm Freundlich**		
qm (mg/g)	**K_L (L/mg)**	**R^2**	**n**	**K_F (mg/g)**	**R^2**
3,7594	**0,0194**	**0,9646**	**126,5823**	**13,7373**	**0,9680**

Based on table 2 through the Langmuir isotherm model, the maximum adsorption capacity (qm) was obtained, which was 3.7594 mg/g with a coefficient value of 0.0194. The qm constant indicates the maximum adsorption power, where the greater the qm value in the Langmuir equation indicates that the adsorption capacity is also greater (Miri, 2022). The Langmuir isotherm is based on three assumptions: The sorption process is limited to one layer (monolayer). All surface sites are similar and can only accommodate one adsorbed atom. The ability of a molecule to adsorb on the appropriate site is unaffected by vacancies on neighboring sites (Zhao, 2011). From the Freundlich isotherm equation, the value of K_F was obtained as 13.7373 mg/g with an n value of 126.5823. The n-value, which is used to assess the degree of truth of an adsorption, is a measure of deviation from the linearity of adsorption and an indicative of a change in the intensity of the adsorption process. Adsorption is supported by the number of n, and the preferred adsorption method is indicated by a value of $n > 1$. The greater the value of the n parameter of the equation, the more heterogeneous a system is (Ismadji, 2021).

CONCLUSION

The highest yield results were obtained in the sample of 90 minutes of extraction variation and 80°C of extraction temperature, namely a pectin yield of 42.5% with a pectin weight of 17 grams from 40 grams of orange peel sample weight and the findings of the study showed that the longer the time and the higher the extraction temperature, the more pectin yield was obtained. The outcomes of the investigation showed that with the addition of pectin mass of 90 mg, the adsorption efficiency of Fe metal in $FeCl_3$ solution for Pacitan Sweet Orange peel was 60.52% and the appropriate isothermal to be used in this study was the Freundlich isothermal where the determination coefficient (R^2) was 0,968. The temperature and drying time of pectin should be further investigated so that the pectin product obtained has the right shape when commercialized. Further research should be conducted with other heavy metals to evaluate the adsorption capacity of pectin.

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