



## Adsorption Performance of Low-cost Java Plum Leaves and Guava Fruits as Natural Adsorbents for Removal of Free Fatty Acids from Coconut Oil

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### PAPER INFO

#### Paper history:

Received 29 April 2019

Received in revised form 04 August 2019

Accepted 13 September 2019

#### Keywords:

Java Plum Leaves

Guava Fruits

Adsorption

Freundlich and Langmuir Isotherms

### ABSTRACT

This study assesses the adsorption performance of Java plum leaves and guava fruits based adsorbents as natural products widely available in Aceh, Indonesia. These renewable adsorbents were employed to remove free fatty acids (FFAs) that cause the rancid odor in coconut oil. The adsorption tests were carried out at three different doses (50, 75, 100 g) and seven agitating periods (1, 2, 3, 4, 5, 6, 7 h). The adsorbents were characterized by Scanning Electron Microscopy (SEM) to observe their morphologies, and Fourier transform infrared (FTIR) spectroscopy to investigate the functional groups. The adsorption kinetics were also analyzed using the Freundlich and Langmuir isotherm models. The SEM image showed that the particle sizes of the guava fruits based adsorbent were 30-45  $\mu\text{m}$  while those of Java plum leaves based adsorbent were 7-15  $\mu\text{m}$ , both showing attractive range to enhance surface area for adsorption sites. FTIR spectra showed the presence of methylene, aliphatic and phenolic groups for both adsorbent, aromatic and alkene groups only for java plum based adsorbent and secondary amine and alcohol groups only for guava fruit based adsorbent. Those groups seem to play important role in enhancing chemical adsorption of FFAs from the coconut oil sample. The results showed that Java plum leaves and guava fruits based adsorbents had a respective maximum adsorption capacity of 144.99 and 133.77 mg/g, with an optimum agitation time of 6 hour. The high absorption capacity could be ascribed from phenolic and flavonoid compounds present in both materials. Kinetics of adsorption of FFAs on both materials obeyed the Freundlich isotherm model indicating a multilayer and heterogeneous surface of adsorbent.

doi: 10.5829/ije.2019.32.10a.06

## 1. INTRODUCTION

Coconut fruits thrive along the coastal area of Aceh province, Indonesia. They are commonly processed as coconut oil and Pliek U, an Aceh traditional food. The locals generally produce coconut oil in three ways. It is done by evaporating coconut water to yield oil as residue, by cooking coconut milk that is produced from the coconut meat, and by pressing the decaying coconut meat until the oil comes out. All of the methods cause a distinct rancid odor in the coconut oil product, while the first, second and third methods lead to a slight, medium and strong rancid odor, respectively. The quality of coconut oils is affected by several parameters. One of them is the

content of free fatty acids (FFAs). FFAs are the result of fat hydrolysis or oxidation reaction during the coconut fermentation process. Hydrolysis reaction occurs due to the interaction between the water and fat, which causes some breaking of the fatty acids from the oil, thereby releasing the FFAs and glycerol [1]. An excess concentration of FFAs can decrease the quality of oil, causing a rancid odor which is harmful to consume continuously, and, hence, lowers the selling price. FFA content formed during the processing of coconut oil can reach 12.8% [2,3].

In order to reduce the content of FFAs in coconut oils, several techniques have been investigated: neutralization, extraction using alcohol, and steam distillation.

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However, these methods have several disadvantages, such as the high energy and solvent consumptions. The neutralization process has some limitations because the glycerides can also be saponified by alkali. Extraction by alcohol releases a considerable amount of solvent and leads to many stages in the process due to the limited solubility of FFAs. Steam distillation needs a high temperature and requires a large amount of energy with poor efficiency [4]. Therefore, adsorption is seen as a promising way of reducing the FFAs contents of oils.

Adsorbent materials can attract specific substances and facilitate their separation from a mixture. Good adsorbents typically pose the following properties: have high mechanical strength, chemical stability, high surface area, proper specific pore structure, and are highly selective and reusable. Some of the adsorbent materials frequently used are activated carbon, synthetic zeolites, nanozeolite clinoptilolite, modified porous silica, natural adsorbent from biomass, graphene oxide manganese ferrite, natural clays, chitosan, and macroporous polymeric adsorbents [5–9].

In recent studies, some researchers have investigated the synthesis methods and assessed the performance of natural agricultural materials as low-cost adsorbents. They are widely available and environmentally benign. The use of natural adsorbents offers two main advantages. Firstly, they reduce the volume of the waste, and secondly, they lower the cost of the adsorbent [10]. The major constituents of agricultural products are usually lignin and cellulose, which are rich in various polar functional groups like alcohols, aldehydes, ketones, carboxylic, phenolic and ether [11].

Many agricultural-based resources have been utilized as biosorbents with promising performance. For instance, Tavlieva et al. [12] investigated the performance of white rice husk in adsorbing brilliant green in aqueous solutions. The results revealed that the white rice husk adsorbent has an acceptable performance with adsorption capacity of up to 85.56 mg/g at 47 °C. Yadav et al. [6] studied the potential of sweet lemon (*Citrus limetta*) waste and rice husk. The performance was evaluated by determining the maximum phosphate removal in batch-wise adsorption. They obtained a satisfactory removal efficiency of phosphate of 95.85% at 24.85 °C using 3 g/l of activated sweet lemon waste adsorbent, at pH 6.0. This excellent performance was attributed to the presence of functional groups in the biosorbent that enhances the affinity towards phosphate. Postai et al. [13] assessed methylene blue (MB) and rhodamine B (RhB) removals using the seeds of *Aleurites moluccana*. The maximum adsorption capacities of MB and RhB reached to 178 and 117 mg/g, respectively.

Reduction of free fatty acids in crude soybean oil with rice hull ash (RHA) has been investigated by Yoon et al. [14], where thermal and acid treatment was carried out before being used as an adsorbent. The results of

adsorption process showed an FFA allowance of 30-40% at 5 g/100 g oil of RHA500. RHA500 was the result of pyrolysis process at a temperature of 500 °C, while the effect of acid activation on RHA was not significant. Ismaila et al. [15] examined the ability of modified potato and cassava starch to reduce FFA levels in biodiesel. The results of the study showed a reduction in FFA by 69.46%. The highest adsorption capacity of modified cassava and potato starch was 233 and 228 mg of FFA per gram of adsorbent at 0:25: 1 KOH / starch molar ratio. Use of silica from rice husk to reduce FFA levels has also been studied [16,17]. Clowutimon et al. [16] reported that the highest obtained adsorption capacity was 185 mg FFA per gram of adsorbent, while the results of the study by Narachai et al. [17] showed the highest adsorption capacity was 277.8 mg/g.

This study assesses the potential of local agricultural resources— Java plum leaves and guava fruits— as adsorbents to remove FFAs from coconut oil. The guava fruits are widely available locally, and often in oversupply, have almost no economic value and are left spoiled during the harvest season. On the other hand, java plum leaves are also widely available around the coconut oil plantation and processing sites and the utilization of the leaves is so far less known. This study explored the utilization of guava fruits and java plum leaves as potential adsorbent material for FFA removal from coconut oil. In this study the effect of the adsorbent mass and agitation time on the performance of FFAs adsorption was investigated. The characterization of the adsorbent was performed using Fourier transform infrared (FTIR) and scanning electron microscopy (SEM). The adsorption kinetics and performance for the removal of FFAs are also discussed.

## 2. MATERIALS AND METHODS

**2. 1. Materials** Java plum leaves and guava fruits as natural adsorbents were collected locally in Aceh. Coconut oil as a sample test for FFA adsorption was purchased from the local market. Ethanol and n-hexane (Merck, Germany) were used as solvents. NaOH (85%, Sigma Aldrich) was used as the titration solution. Phenolphthalein (PP, Merck, Germany), was used as an equilibrium indicator for alkali titration.

### 2. 2. Preparation and Characterization of Adsorbent

About 100 g of sliced Java plum was dried in the oven at 50° C overnight to reduce the moisture content until a constant weight of the materials was obtained. The dried guava fruit was further processed using a laboratory mill 120 (Perten Instruments, Sweden) for 1 hour to obtain a powder size of 100 mesh. The same treatment was also applied for the guava fruit.

**2. 3. SEM Analysis** The morphology of the fabricated adsorbents was analyzed using a SEM (JSM-6510LA–Japan). The powder was placed on top of carbon tape and was coated with gold (about 10 nm thickness) to induce conductive properties. The analysis was found best at voltage of 15 kV and the magnification was set to obtain representative images.

**2. 4. FTIR Analysis** The surface chemical compounds of both adsorbents were investigated using FTIR. The powder sample was mixed with potassium bromide 10:90 w/w (Fluka) and mechanically pressured to form transparent thin film suitable for the FTIR analysis. The IR spectra absorption for each adsorbent was obtained using a Shimadzu IRPrestige-21. The data were recorded at room temperature in the wavelength range of 4000 – 400  $\text{cm}^{-1}$ .

**2. 5. Adsorption Capacity** Java plum at different weights of 50, 75, and 100 g was added into 300 ml of coconut oil separately. The mixtures were stirred using a magnetic stirrer for seven different adsorption periods (1, 2, 3, 4, 5, 6, and 7 hours). The formed slurry was filtered using filter paper (Whatman no. 40). Then, the filtrate was titrated to determine the remaining FFAs. The adsorption capacity of Java plum leaves and guava fruits at equilibrium was calculated using Equation (1).

$$q = \frac{(C_0 - C_e)}{m} \times V \quad (1)$$

where  $q$  is the capacity of adsorption (mg/g);  $C_0$  is the initial concentration of FFAs in coconut oils (mg/ml);  $C_e$  is the equilibrium concentration of FFA (mg/ml);  $V$  is the volume of coconut oil (ml); and  $m$  is the dose of the adsorbent used (g).

## 2. 6. Adsorption Equilibrium Isotherm

**2. 6. 1. Freundlich Adsorption Isotherm** The Freundlich isotherm describes the reversible adsorption with no limitation in terms of monolayer formation. This empirical model can be adopted to multilayer adsorption, with the non-similar distribution of adsorption heat and affinities throughout a heterogeneous surface [18]. The Freundlich adsorption isotherm is expressed in Equations (2) and (3).

$$q_e = K_f \times C_e^n \quad (2)$$

or as a linear equation: 1

$$\log q_e = \log K_f + n \cdot \log C_e \quad (3)$$

where  $q_e$  is the quantity of adsorbed FFAs at equilibrium state (mg/g);  $C_e$  is the concentration of FFAs in the aqueous phase at equilibrium (ppm);  $K_f$  (L/g) and  $n$  are the Freundlich constants which correlate with the capacity and intensity of adsorption, respectively.

**2. 6. 2. Langmuir Adsorption Isotherm** The Langmuir adsorption isotherm is originally extended to describe the adsorption of the gas-solid phase on the adsorbent. This model is based upon two hypotheses; namely, no interaction forces between the adsorbed molecules and no further sorption takes place once a molecule fills a surface [18]. The Langmuir isotherm in a linear equation is described in Equation (4):

$$\frac{C_e}{q_e} = \frac{K}{q_m} + \frac{1}{q_m} C_e \quad (4)$$

where  $q_e$  is the amount of FFAs adsorbed at equilibrium (mg/g);  $C_e$  is the concentration of FFAs in the aqueous phase at equilibrium (ppm).  $q_m$  is the maximum adsorption of FFAs (mg/g), and  $K$  is the Langmuir constant related to the adsorption capacity and the energy of adsorption (g/mg).

**2. 7. Analysis** The content of the FFAs and water in the oil were measured before and after adsorption treatment. The amount of FFAs in the oil was measured using alkali titration in an alcohol solution. A weighed amount of 2.5 to 3 g of oil was added into a flask and dissolved in 10 ml of n-hexane, followed by the addition of 15 ml ethanol. Two drops of 0.1% phenolphthalein (pp) were added into the mixture as an indicator. The solution was then titrated by 0.1 N NaOH solution. The titration was stopped when the orange color solution was constantly displayed. The concentration of FFAs was calculated using Equation (5).

$$\text{FFA content} = \frac{V \times N \times 54.91}{W} \times 100\% \quad (5)$$

where 54.91 is the molecular weight of the coconut oil (g/mol),  $V$  is the volume of NaOH as the titrant solution (ml),  $N$  is the concentration of NaOH (N), and  $W$  is the mass of the oil (g). The calculation result gives FFA content (%) which is then converted into units of mg/mL.

The water content in the oil was analyzed to determine the quality of the oil. 10 g oil was stirred until homogenous and poured into the porcelain cup. The porcelain cup was placed in the oven at 105 °C for 3 hours. The mass of the sample was weighed before ( $m_0$ ) and after heating ( $m_e$ ) until a constant mass was obtained. The water content in the oil was calculated using Equation (6).

$$\text{Water content} = \frac{m_0 - m_e}{m_0} \times 100\% \quad (6)$$

## 3. RESULTS AND DISCUSSION

**3. 1. Morphology of the Adsorbent** Natural adsorbents from Java plum leaves and guava fruits after size reduction are shown in Figure 1. Adsorbent morphologies are used to identify the size distribution of

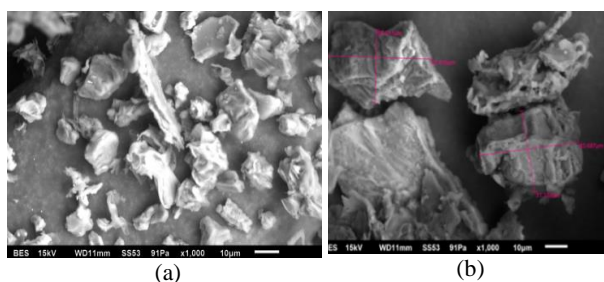
the particles and the typical shape of the adsorbent. The micro-scale images (magnification 1000x) showing the morphology of adsorbents are presented in Figure 2. Java plum leaves and guava fruit particles have a heterogeneous form, largely rectangular in shape. The particle sizes of the guava fruits are generally larger than that of the Java plum leaves. The average size of the guava fruit particles is 30-45  $\mu\text{m}$ , while Java plum leaves have a particle size of approximately 7-15  $\mu\text{m}$ . The final particle sizes were not only affected by the treatment process (milling) but also the nature of the materials.

The java plum leaf are constructed of aggregates of small size particulates, while the guava fruits are from larger aggregates. The milling process was actually to reduce the size to ease processing and did not affect the microstructure of the resulting particles. The particle size distributions have a great impact on the adsorption activity. A smaller particle size offers a larger active surface area that supports high adsorption capacity.

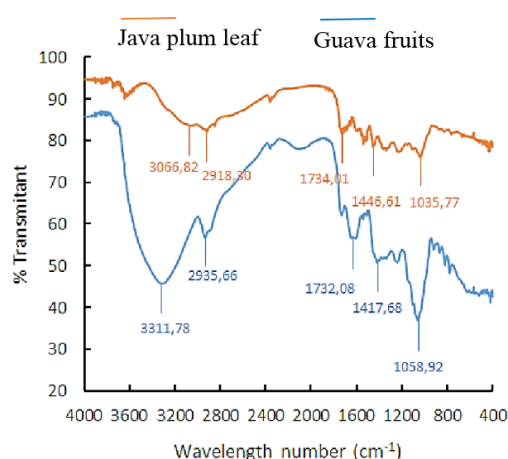
**3. 2. FTIR Spectroscopy** The FTIR spectra of both Java plum leaves and guava fruits are shown in Figure 3. FTIR analysis is used to investigate the chemical compounds in the Java plum leaves and guava fruits as natural adsorbents. The spectra were collected at a wavelength range of 4000– 400  $\text{cm}^{-1}$  and were recorded as transmittance values. The spectra in Figure 3 show that both natural adsorbents have similar peak locations (wavelength number). Each peak interprets the chemical bond contained in the adsorbent.



**Figure 1.** Adsorbent powder from (a) Java plum leaves and (b) guava fruits



**Figure 2.** SEM images of adsorbent from (a) Java plum leaves and (b) guava fruits



**Figure 3.** FTIR spectra of natural adsorbents

Identification of the functional groups for Java plum leaves is tabulated in Table 1. A weak peak at 3066  $\text{cm}^{-1}$  relates to the aromatic group with C–H stretch. The same stretch is also found in the peak at 2918  $\text{cm}^{-1}$ , which indicates the methylene group. The C=O stretching vibration in the aliphatic aldehyde group showed at a peak at 1734  $\text{cm}^{-1}$  and indicates lipid and flavonoid contents [19]. At 1446  $\text{cm}^{-1}$ , the C=C stretch corresponds to the alkene group. The phenolic group as a prominent component presents at 1035  $\text{cm}^{-1}$ . Based on several functional groups obtained by the FTIR spectra, phenolic compounds and flavonoids contained in java plum leaves that play an important role in the adsorption of FFA. Phenolic compounds consist of one or more aromatic rings and at least one hydroxyl function directly linked to the rings [20]. Hydrogen in these two active compounds bonds with FFA which affects the reduction of FFA levels in coconut oil.

The identification of peaks for guava fruits is shown in Table 2. The N-H stretch of the amine group at a peak location of 3311  $\text{cm}^{-1}$  indicates the amino acids component. At 2935  $\text{cm}^{-1}$ , the C-H stretch of the methyl group is observed. The same components as the Java plum leaves present in the spectrum of guava fruits; C=O stretch of the aliphatic aldehyde group is also found in

**TABLE 1.** Interpretation of chemical groups using FTIR on Java plum leaves [18]

No	Wavenumber ( $\text{cm}^{-1}$ )		Band	Functional group
	Java plum leaves	Literature		
1.	3066.82	3100-2990	C-H	Aromatic
2.	2918.3	2940-2860	C-H	Methylene
3.	1734.01	1740-1720	C=O	Aliphatic aldehyde
4.	1446.61	1600-1430	C=C	Alkene
5.	1035.77	1260-1000	C-O	Phenol

**TABLE 2.** Interpretation of chemical groups using FTIR on guava fruits

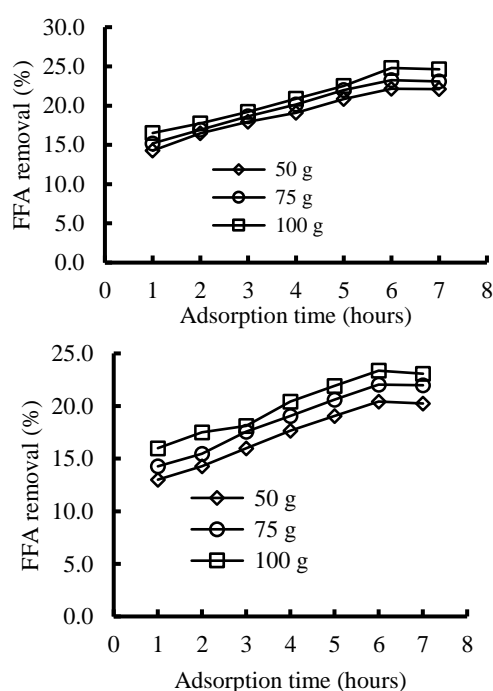
No	Wavelength number (cm <sup>-1</sup> )		Band	Functional group
	Guava fruits	Literature		
1.	3311.78	3300-3310	N-H	Secondary Amine
2.	2935.66	2940-2860	C-H	Methylene
3.	1732.08	1740-1720	C=O	Aliphatic aldehyde
4.	1417.68	1420-1330	O-H	Alcohol
5.	1058.92	1260-1000	C-O	Phenol

the spectrum of this sample. The aldehyde group generates flavor molecules in plants [18]. The O–H bonding and C–O stretching vibrations that showed peaks at 1417 and 1058 cm<sup>-1</sup>, respectively, correlate with the alcohol and phenol groups due to the presence of flavonoid compounds [18]. The results of FTIR spectra show that there are several functional groups which indicate the presence of phenolic compounds and flavonoids. These two active compounds influence the adsorption of FFA from coconut oil. Out of all functional groups available, aliphatic aldehydes group found in both adsorbents are known to be highly effective in increasing the total pore volume and thus the active adsorption sites [21].

**3. 3. Oil Characteristics** The quality of coconut oil measured in this experiment included FFAs and moisture content. The results showed that the FFA content was 19.1% and the water content was 0.7%. According to the Quality Standard of Oil Based on Coconut (SNI 01-2902-1992), the value of FFAs and water content in the oil was greater than the standard limit. The maximum contents for both parameters are 5 and 0.5%, respectively.

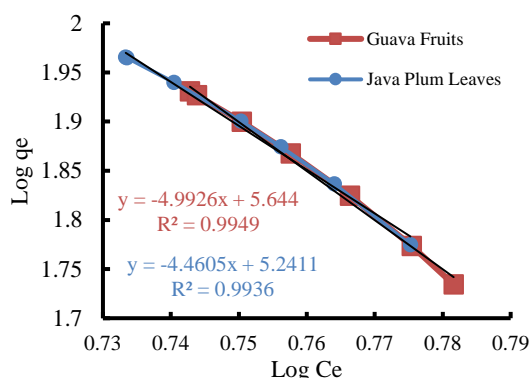
**3. 4. Effect of Adsorption Time and Adsorbent Dose** The adsorption of FFAs from coconut oil was affected by the amount of adsorbent and the time of adsorption. The results of the adsorption experiments are shown in Figure 4. The percentage of FFAs removed increased as a function of time. The longer the time, the more were the adsorbents in contact with the oil, and the more would be the FFA molecules adsorption.

Both adsorbents showed the best adsorption performance and finite adsorption capacity at 6 hours agitating. The process reached an equilibrium where no more adsorption occurred at agitation time of 6 hours. Extending the contact time to 7 hours concluded to desorption. The mass of adsorbent also plays an important role in the adsorption activity, where a higher concentration of adsorbent has greater potential to lead to higher adsorption capacity. The best amount to remove FFAs for both natural adsorbents is at 100 g. The adsorption process using Java plum leaves and guava fruits showed the FFAs removal efficiencies of 24.8 and 23.1%, respectively.

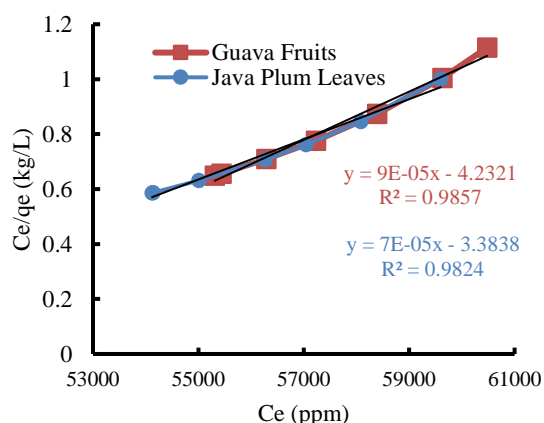
**Figure 4.** The effect of contact time and adsorbent amount on the removal of FFAs using (a) Java plum leaves and (b) guava fruits

**3. 5. Adsorption Capacity** Both adsorbents gave the best adsorption performance with a mass of 100 g of adsorbent and 6 hours contact time. The adsorption capacity in this study is the amount of FFA adsorbed per gram of adsorbent. However, the Java plum leaves showed better adsorption activity for the removal of FFAs than the guava fruits. The comparison of the adsorption capacity between the Java plum leaves and guava fruits is shown in Figure 5. Java plum leaves reaches an adsorption capacity of up to 141.99 mg/g, while the guava fruits had a lower capacity of 133.77 mg/g. The reason for this performance is related to the adsorbent size. The Java plum leaves have a smaller particle size than guava fruits, so they have a larger surface area. The surface area allows the adsorbent to adsorb more molecules and enhance the capacity of adsorption.

**3. 6. Adsorption Isotherm** The adsorption kinetics of Java plum leaves and guava fruits follows Freundlich and Langmuir isotherm. Based on the Freundlich and Langmuir isotherm graphs showed in Figures 5 and 6, the R<sup>2</sup> for both adsorbents is approximately 0.99 for the Freundlich isotherm. The R<sup>2</sup> value of the Langmuir isotherm curve for the Java plum leaves and guava fruits is 0.98. The model hypothesized that the adsorption activity occurs in multilayer and on heterogeneous surface.



**Figure 5.** Freundlich adsorption isotherm for Java plum leaves and guava fruits based adsorbent



**Figure 6.** Langmuir adsorption isotherm for Java plum leaves and guava fruits based adsorbent

Adsorption isotherms are important in the investigation of adsorption mechanism. The main adsorption characteristics, such as surface property, adsorbent affinity, and the maximum adsorption capacity can be evaluated by the adsorption isotherm and related constants [18]. The experimental adsorption isotherms were measured at 293.15 K. In order to correlate this experimental adsorption data, the Freundlich and Langmuir isotherm equations were used.

#### 4. CONCLUSIONS

The natural adsorbents, Java plum leaves and guava fruits showed high potential for the removal of FFAs. According to the FTIR analysis, both adsorbents consisted of phenolic and flavonoid compounds. The percentage of FFAs removed using Java plum leaves reached up to 24.8% with a maximum adsorption capacity of 141.99 mg/g, while guava fruits yielded in a removal efficiency of 23.058% of FFAs with an adsorption capacity of 133.77 mg/g. The better

adsorption capacity of the Java plum leaves was affected by their smaller particle size (7-15 $\mu$ m), compared with guava fruits (30-45  $\mu$ m). The smaller size of adsorbent provided a larger active surface area that provided better adsorption activity. Results show that 6 hours agitating is optimum for both the Java plum leaves and guava fruits. Furthermore, the equilibrium adsorption isotherm for both adsorbents obey the Freundlich isotherm models indicating a multilayer adsorption and heterogeneous surface of adsorbent.

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### PAPER INFO

چکیده

#### Paper history:

Received 29 April 2019

Received in revised form 04 August 2019

Accepted 13 September 2019

#### Keywords:

Java Plum Leaves

Guava Fruits

Adsorption

Freundlich and Langmuir Isotherms

این مطالعه به بررسی عملکرد جذب برگهای آلو جاوا و میوه گواوا به عنوان جاذبهای طبیعی که به وفور در اندونزی یافت می‌شوند، می‌پردازد. از این جاذبهای تجدیدپذیر برای حذف اسید چرب آزاد که باعث ایجاد ترشیدگی در روغن نارگیل می‌شوند، استفاده شد. آزمایشهای جذب با سه دز مختلف جاذب (۷۵، ۱۰۰ و ۱۰۰ گرم) و زمانهای اختلاط مختلف (۱، ۲، ۳، ۴، ۵، ۶ و ۷ ساعت) انجام شد. جاذبها با میکروسکوپ الکترونی روشی مورد بررسی قرار گرفتند تا مورفولوژی آنها بررسی شود و از آنالیز FTIR برای تعیین گروههای عاملی سطحی استفاده شد. سینتیک جذب همچنین توسط ایزوترمهای جذب لانگمایر و فروندلیچ بررسی شد. تصاویر SEM نشان دادند که اندازه ذرات میوه گواوا و برگهای آلو جاوا به ترتیب ۴۵-۳۰ و ۱۵-۷ میکرون بود. نتایج FTIR مویید حضور متیلن، گروههای آلیفاتیک و فنولیک در هر دو جاذب، گروههای آروماتیک و آلکن در جاذب برگ آلو جاوا و آمین نوع دوم و الکل در جاذب میوه گواوا بود. این گروهها نقش مهمی در افزایش جذب اسیدهای چرب آزاد از روغن نارگیل دارند. حداکثر میزان جذب حاصل از برگهای آلو جاوا و میوه گواوا به ترتیب ۱۴۴/۹۹ و ۱۳۳/۷۷ میلی گرم به گرم در زمان ۶ ساعت بود. این جذب بالا را می‌توان به حضور ترکیبات فلاونوئیدی و فنولی در هر دو جاذب نسبت داد. سینتیک جذب برای هر دو جاذب از مدل فروندلیچ پیروی کرد که نشان دهنده جذب چند لایه و سطح هتروژن جاذبها بود.

doi: 10.5829/ije.2019.32.10a.06