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Durian Waste Husks as an Adsorbent in Improving Soaking Water during the Retting Process of *Piper nigrum* L. (Pepper Berries)

Muhammad Hazwan Hamzah [1,2,*], Ainaa Syaheera Amran [1], Adila Fazliyana Aili Hamzah [1], Mohd Zuhair Mohd Nor [3], Rosnah Shamsudin [3], Hasfalina Che Man [1,2] and Wan Aizuddin Wan Razali [4]

1 Department of Biological and Agricultural Engineering, Faculty of Engineering, Universiti Putra Malaysia, Serdang 43400, Malaysia
2 SMART Farming Technology Research Centre, Faculty of Engineering, Universiti Putra Malaysia, Serdang 43400, Malaysia
3 Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, Serdang 43400, Malaysia
4 Faculty of Fisheries and Food Science, Universiti Malaysia Terengganu, Kuala Nerus 21030, Malaysia
* Correspondence: hazwanhamzah@upm.edu.my; Tel.: +60-397696422

Abstract: The potential of raw durian husk and NaOH-modified durian husk as an adsorbent, using different doses, 0.5 g, 1.0 g, 1.5 g, and 2.0 g, is investigated to improve soaking water of pepper berries during the retting process. The surface area and the pore size of the durian husk were examined using Brunner Emmett and Teller analysis. The surface area of NaOH-modified durian husk is higher (2.33 m^2/g) compared to the raw durian husk (1.51 m^2/g). NaOH-modified durian husk has a higher porous structure than the raw durian husk, but both pore diameters are more than 50 nm, which is considered micropore raw material. The effect of the raw durian husk on pH, chemical oxygen demand (COD), dissolved oxygen (DO), and turbidity were compared to the NaOH-modified durian husk with different doses. The 2.0 g of NaOH-modified durian husk enhanced changes in the four parameters. The highest pH value using NaOH-modified durian husk was 6.10 ± 0.02, while turbidity and COD increased to 971.33 ± 1.15 NTU and 1984.67 ± 3.21 mg/L, respectively. The DO of NaOH-modified durian husk shows the lowest reduction to 1.49 mg/L with 2.0 g of NaOH-modified durian husk. The experimental data was best fitted with a first-order kinetic model. Durian husk treated with NaOH could be used as a potential adsorbent to enhance the soaking water for pepper berries.

Keywords: durian husk; soaking; adsorbent; retting; pepper berries; kinetic

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1. Introduction

Piper nigrum L. (pepper berry) is widely known as the King of Spices and is the oldest spice globally. Pepper berries are a perennial plant with climbing vines that prefers to grow natively in tropical conditions with temperatures around 23–30 °C, annual rainfall (2000–3000 mm), relative humidity (70–90%), soil pH (5.5–6.5), and with good soil structure, good water holding capacity, and well drainage [1]. Malaysia is one of Southeast Asia's top producers of pepper berries due to its favourable climate. The common pepper berries widely produced in Malaysia are white and black pepper. In recent years, pepper has been used in food-related industries, traditional medicine, and beauty care [2]. Currently, the demand for white pepper has increased compared to black pepper due to the mild flavour, pungent aroma, and light colour [3]. The process for white pepper is obtained from dried seeds of pepper berries after removing the pericarp [4]. In order to obtain the white pepper, the fresh berries require different processes to decorticate the pepper's outer pericarp. The most common practices in producing white pepper are via the water retting process. Prior studies by Sarawak and Johor have introduced two conventional methods in the retting process: soaking in flowing water and stagnant water [5–7].

The retting procedure uses natural microorganisms to biologically loosen and remove the pericarp of pepper berries. The process takes two weeks of soaking to make the tough and thick skin of the pepper soften and peel off easily [4]. These are the conventional methods for making white pepper in Malaysia; nonetheless, the methodology has significant drawbacks. A prior study discovered that the process approach is unclean due to the retted scent in the white pepper created by the retting process. The process is unhygienically caused by anaerobic bacterial fermentation, producing a rotten smell [8]. In addition, the pepper berries will decay for a week due to contact with flowing or stagnant water during the retting process. The pepper skin will dissolve in water, contaminate the water, and change the colour of the water to black. Therefore, if a large volume of soaking water is discarded into natural watercourses such as rivers after sufficient retting has been performed, this can cause water pollution that harms the environment.

The quality of soaking water during the retting process of pepper berries was a major environmental problem. Research conducted by Megat Ahmad Azman et al. [5] consistently showed that a soaking water test affects the properties of water quality. The method used for the soaking test was separating water into three tanks with different quantities of pepper berries and observing for seven days without water changes. The results indicate that the tank with the highest quantities of pepper berries had the highest increments of turbidity and chemical oxygen demand (COD) and the highest reduction of pH value and dissolved oxygen (DO). Other research methods for the retting process of pepper berries include utilizing enzymes or microbes [7,9–11], flowing water [6], and ozone [8]. However, no previous study has investigated the retting process of pepper berries with different doses of adsorbent in improving the soaking water.

In this study, an adsorbent has been suggested to improve the quality of soaking water during the retting process of pepper berries and to shorten the retting period. Recently, the utilization of biological material as biosorbents from agricultural waste has received greater attention. Being cost-effective and renewable, many types of biomass waste from different parts of plants have been utilized as an adsorbent to remove a wide range of organic and inorganic dissolved pollutants from water and air [12]. For example, agricultural wastes such as hazelnut shells, orange peels, maize, peanut shells, and jackfruit in natural or modified forms have been explored and have significant removal efficiency [13]. The agricultural wastes are also natural, inexpensive, non-toxic, biodegradable, and eco-friendly, making them appropriate for environmental treatment or chemical separation [14].

In that context, durian husk is also a typical example of biomass that has been considered a biosorbent in this study. Durian husk was chosen as a potential adsorbent for improving the soaking water during the retting process. Durian (*Durio zibethinus*) is the king of fruits native to Southeast Asia, widely produced in Thailand, Malaysia, the Philippines, and Indonesia [15]. It is a seasonal fruit in tropical Asia. Usually, durian is consumed fresh by eating flesh; consumers can eat only half of the fruit from the whole mass due to the fruit's characteristics. Due to the high consumption, large amounts of durian husk are disposed of, posing a threat to the environment, particularly given the limited space available at landfill sites today to decompose the waste [16]. Thus, the resultant quantity of durian waste accounts for the feasibility of selecting it as a biosorbent in this study.

Durian husk has been reported in the treatment of several organic and inorganic pollutants due to its lignocellulosic properties [17]. For instance, durian husk was applied for the adsorption of methylene blue in water [18] and Fe(II) ions adsorption from an aqueous solution [19]. Physical and chemical activation of biomass such as durian husk is essential to improve sorbate removal from contaminated water. Furthermore, adsorbents derived from agricultural waste are employed in their original state or with physical or chemical modification as a pre-treatment. Adsorbents are pre-treated to remove all colours, metals, and soluble organic compounds from aqueous solutions and improve their adsorption capacity [20]. Depending on the type of adsorbate or pollutant to remove, different adsorbents are treated with different modification agents, such as organic compounds, minerals, dyes, acid and base solutions, calcium hydroxide, sodium carbonate, and sodium hydroxide to increase the efficacy of removal process.

Overall, the retting method is time-consuming and labour-intensive because pericarp removal may take longer due to a decrease in DO in water, which is undesirable since decreasing oxygen would postpone the preparation of white pepper. Furthermore, white pepper is in short supply in Malaysia, so solving the issues can increase pepper production. Therefore, durian adsorbent is expected to minimize the retting time for white pepper production, prevent water pollution, and provide the highest pepper quality. In this study, the kinetic study analysis was carried out to express the numerical variations in prediction quality of soaking water during the retting process [5]. Overall, this study aims to determine the potential of durian husk as an adsorbent in improving soaking water during the retting process of pepper berries. The following are the specific objectives of this study: (a) to determine the physical characteristics of the durian husk as an adsorbent, (b) to determine the effect of adsorbent dose on the quality of soaking water during the retting process of pepper berries, and (c) to determine the kinetic models of the soaking water during the retting process of pepper berries.

2. Materials and Methods

2.1. Flowchart of Methodology

The overall flowchart of the methodology is illustrated in Figure 1.

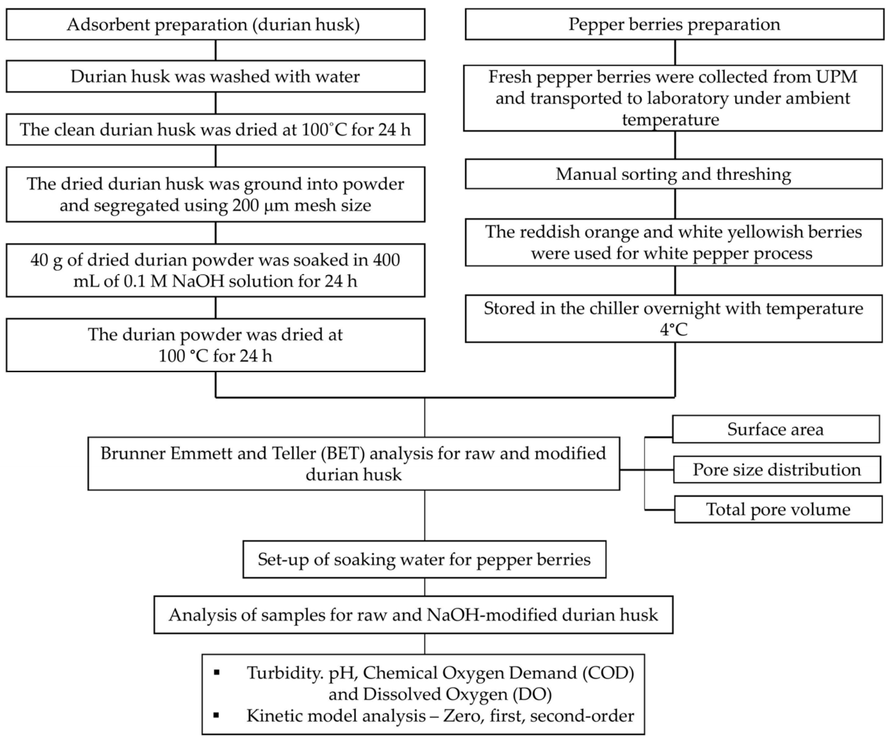

Figure 1. Flowchart of methodology.

2.2. Preparation of Raw and NaOH-Modified Adsorbents from Durian Husk

Durian husk was collected from the local market in Negeri Sembilan. The variety that was selected is the durian D24 variety. The NaOH-modified adsorbent was prepared with modification according to the method developed by Gan and Cheok [21]. First, durian husk was washed with water to remove dirt on its surface. Following this process, the clean sample was dried using an oven (MEMMERT Universal Oven UN55, Bavaria, Germany) at 100 °C for 24 h. Next, the dried sample was ground using a grinder machine (PH-1, Xinganbangle, Yiwu, China) to produce durian adsorbent powder and segregated using

200 µm mesh size. Next, 40 g of dried durian powder was soaked in 400 mL of 0.1 M NaOH solution for 24 h. Then, 3.2 g of NaOH pellets were dissolved in 800 mL of distilled water to make a 0.1 M NaOH solution. After 24 h of soaking, durian husk powder was filtered and rinsed multiple times with distilled water. The rinsed powders were then dried in a 100 °C oven for 24 h. The NaOH-modified adsorbents were labelled and stored in an airtight container.

2.3. Brunauer–Emmett–Teller (BET) Surface Area Analysis

The BET surface area analysis of raw and NaOH-modified durian husk was performed using the SA-9600 BET surface area analyser before the retting process [22]. The BET analysis focused on the durian husk's surface area and pore volume based on the nitrogen adsorption–desorption process at 77.268 K.

2.4. Pepper Berries Sample Preparation

The pepper berries were prepared following method described by Megat Ahmad Azman et al. [5]. Fresh mature pepper berries were obtained from Putra Agriculture Centre, Universiti Putra Malaysia. The pepper berries were manually sorted and threshed to remove leaves and spikes. The mature pepper berries were delivered to the laboratory. The reddish-orange and white-yellowish berries were used for white pepper processing. Then, the berries were stored in the chiller overnight at 4 °C.

2.5. Experimental Set-Up of Soaking Water for Pepper Berries

Figure 2 shows the experimental set-up of soaking water for pepper berries. The following method was adapted and modified from Megat Ahmad Azman et al. [5]. A jute gunny bag with dimensions of 10 cm × 6 cm × 3 cm was filled with 200 g of pepper berries. Then, the jute gunny bag that contained pepper berries was immersed in the soaking tank. The soaking tank was filled with 1400 mL. The tank was filled with 0.5 g of adsorbent, and the soaking water was monitored for 7 days. An amount of 200 mL of soaking water was sampled daily to determine the properties of the soaking water before and after the adsorption process for raw and NaOH-modified durian husk. The sample of soaking water for day one was taken as soon as the pepper berries were soaked in the prescribed conditions. The experiment was conducted in triplicates. The experiment was conducted using both raw durian husk and NaOH-modified durian husk for other amounts of adsorbent (1.0 g, 1.5 g, and 2.0 g).

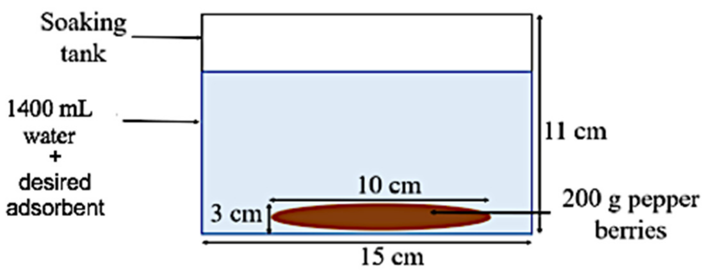

Figure 2. Experimental set-up of soaking water for pepper berries.

2.6. Analysis of Soaking Water Quality

The pH, turbidity, and dissolved oxygen (DO) analysis were subjected to the method explained [6]. The chemical oxygen demand (COD) concentration was determined in accordance with the standard procedure described [23].

2.6.1. pH

The pH value of the soaking water was determined using YSI Professionals Plus Multimeter (Xylem, Washington, DC, USA) at room temperature. This process was repeated every day for 7 consecutive days.

2.6.2. Turbidity

10 mL of water sample from each tank was used to determine the turbidity of water. The turbidity of water was measured using the portable turbidity meter (2100Q, Hach, Loveland, CO, USA). The data was recorded every day for 7 consecutive days.

2.6.3. COD

The COD method was performed for 7 consecutive days using a spectrophotometer (DR/4000U, Hach, Loveland, CO, USA). A 10 mL soaking water sample was added to a 1.5 mL strong oxidant (potassium dichromate solution) and 3.5 mL sulphuric acid, with silver as a catalyst. Then, the sample was placed in the digestor for the digestion reagent process at a temperature of 150 °C for 2 h. After digestion, the sample was cooled down for 10 min before being placed in a spectrophotometer. The COD value of the sample was displayed as mg/L.

2.6.4. DO

The DO concentration was measured using a YSI Professionals Plus Multimeter (Xylem, Washington, DC, USA). The data was collected every day for 7 consecutive days.

2.7. Kinetic Model Analysis

The kinetic of soaking water during white pepper production was studied using zero-, first-, and second-order kinetic models for turbidity, pH, COD, and DO [24]. The determination coefficient (R^2) verified the most accurate kinetic model-fitting analysis. The order of response was carried out at different water quality parameters depending on the soaking period. The following equations were used to express the zero-order, first-order, and second-order kinetic models:

$$\text{Zero-Order} = C = -kt + C_0 \quad (1)$$

$$\text{First-Order} = \ln C = -kt + C_0 \quad (2)$$

$$\text{Second-Order} = \frac{1}{C} = kt + \frac{1}{C_0} \quad (3)$$

where C = the measured value for each parameter of water quality; C_0 = the initial value of the measured parameter of water quality; and k = the rate constant; t = the soaking time.

3. Results and Discussion

3.1. BET Analysis

Table 1 tabulated the BET analysis of raw durian husk and NaOH-modified durian husk.

Table 1. BET analysis of raw and NaOH-modified durian husk.

Adsorbent	Pore Volume (cm^3/g)	Pore Size (nm)	Surface Area (m^2/g)
Raw durian husk	0.28	3660.00	1.51
NaOH-modified durian husk	0.30	2587.00	2.33

Overall, the surface area of NaOH-modified durian husk is higher (2.33 m^2/g) compared to the raw durian husk (1.51 m^2/g). Obviously, NaoH-modified durian husk has a lower pore size than raw durian husk, which caused the surface area to be greater. This is demonstrated by the determined and displayed pore size value in Table 1, as well as the pore volume. This also signified the importance of surface modification of raw agricultural waste prior to adsorption studies [25,26]. When the specific surface area of an adsorbent rises, the number of accessible active sites on that adsorbent increases, resulting in a higher adsorption capacity of removal [27,28].

Moreover, pores in solid particles have several properties, and the easiest visual property of a pore is its size. Additionally, pore size has the greatest influence on the properties of solid adsorbents, such as biomass, compared to other parameters. It is useful and convenient to categorically use pore diameter based on pore size distribution to characterize and compare different porous solids [29]. Evidently, the average pore diameter, as presented in Table 1, showed the decrement of pore diameter from 3660 nm (raw durian husk) to 2587 nm (NaOH-modified durian husk). The significant difference occurred when the adsorbent passed through chemical modification. These findings imply that it lowers pore diameter and gives a more reactive surface area, increasing the reaction rate for chemical processes [30,31]. Since both pore diameters are greater than 50 nm, this adsorbent can be considered macropore material [32]. A further study of the pore size, surface area, and other physico-chemical characteristics can be assessed and validated by different methods in the future, including scanning electron microscopy (morphology), Fourier Transform Infra-Red (chemical composition), and X-ray diffraction (changes in microstructure).

3.2. Analysis of Soaking Water Quality

3.2.1. Effect of Adsorbent Dose on pH

Table 2 presented the effect of adsorbent dose on pH for both raw and NaOH-modified durian husk. Overall, it was observed that the pH value increased with an increasing adsorbent dose using raw and modified adsorbents prepared from durian husk. Megat Ahmad Azman et al. [5] reported that after 7 days for any amount of pepper berries, soaking water has the lowest pH value due to the increasing acidic compound from pepper berries, which can cause water pollution.

In addition, malodours may arise during the retting process as a result of the formation of methylphenol and methylindole [33]. As a result, the soaking water should be treated according to the relevant standard before being discharged into the natural watercourse. Figure 3 shows that 2.0 g of adsorbent for both raw and NaOH-modified durian husk had a lower pH value after 7 days of treatment. Additionally, 2.0 g of adsorbent added to the water increases the adsorption capacity and reduces dissolved organic matter, and it provides additional active sites, oxygen, and functional groups for effective adsorption removal [22]. The reduction of organic matter improves the pH value due to lower acidity.

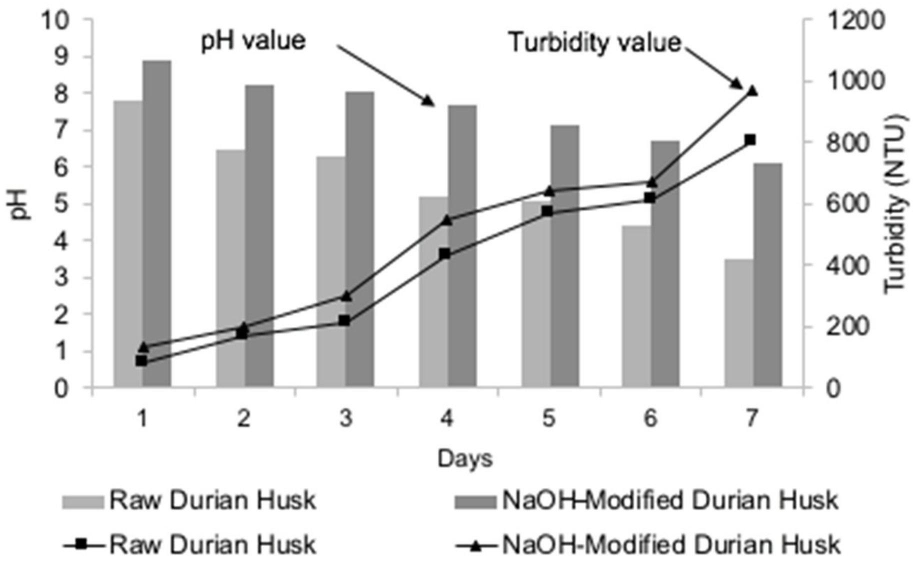

Figure 3. Effect of pH and turbidity value for 2.0 g of raw and NaOH-modified durian husk.

Table 2. Mean value for pH and turbidity of soaking water with raw and NaOH-modified durian husk from day 1 to day 7.

Day	pH							
	Raw Durian Husk				NaOH-Modified Durian Husk			
	A	B	C	D	A	B	C	D
1	7.77 ± 0.02	7.85 ± 0.03	7.85 ± 0.01	7.84 ± 0.02	8.13 ± 0.02	8.57 ± 0.02	8.86 ± 0.05	8.92 ± 0.03
2	6.52 ± 0.01	6.54 ± 0.02	6.52 ± 0.01	6.49 ± 0.03	7.68 ± 0.20	8.00 ± 0.04	8.12 ± 0.01	8.22 ± 0.01
3	6.22 ± 0.01	6.25 ± 0.01	6.26 ± 0.00	6.28 ± 0.02	7.29 ± 0.02	7.90 ± 0.03	7.98 ± 0.01	8.03 ± 0.02
4	5.16 ± 0.02	5.23 ± 0.02	5.23 ± 0.01	5.21 ± 0.01	6.87 ± 0.02	7.81 ± 0.02	7.66 ± 0.02	7.69 ± 0.01
5	5.05 ± 0.01	5.06 ± 0.02	5.07 ± 0.02	5.09 ± 0.02	6.18 ± 0.03	6.96 ± 0.01	7.03 ± 0.04	7.13 ± 0.01
6	4.30 ± 0.02	4.36 ± 0.11	4.39 ± 0.02	4.42 ± 0.01	5.87 ± 0.01	6.35 ± 0.01	6.66 ± 0.03	6.73 ± 0.01
7	3.10 ± 0.02	3.88 ± 0.01	3.77 ± 0.01	4.61 ± 0.02	5.22 ± 0.01	5.85 ± 0.02	5.98 ± 0.05	6.10 ± 0.02
Day	Turbidity (NTU)							
	Raw Durian Husk				NaOH-Modified Durian Husk			
	A	B	C	D	A	B	C	D
1	73.33 ± 1.53	87.00 ± 1.00	86.33 ± 0.58	86.39 ± 1.15	93.00 ± 1.00	102.33 ± 0.58	114.00 ± 1.00	132.67 ± 2.08
2	170.33 ± 2.08	168.33 ± 2.08	165.33 ± 3.21	168.67 ± 0.58	188.00 ± 0.82	197.00 ± 0.82	203.33 ± 2.08	197.67 ± 2.52
3	288.00 ± 3.61	213.00 ± 2.00	213.00 ± 1.00	217.00 ± 2.65	297.67 ± 1.53	276.67 ± 1.15	291.33 ± 1.00	303.33 ± 2.08
4	432.33 ± 1.15	429.00 ± 1.00	428.33 ± 1.53	435.00 ± 3.00	491.00 ± 1.00	520.00 ± 2.00	548.00 ± 4.00	552.00 ± 2.08
5	543.00 ± 2.00	541.00 ± 1.00	541.67 ± 0.58	573.00 ± 1.00	585.00 ± 1.00	610.67 ± 1.53	627.67 ± 3.06	642.00 ± 2.00
6	607.00 ± 1.00	598.00 ± 1.00	604.33 ± 1.15	612.00 ± 1.00	691.00 ± 1.00	653.00 ± 2.65	664.00 ± 2.65	674.00 ± 1.00
7	797.67 ± 0.58	799.33 ±1.53	873.52 ± 0.01	855.00 ± 1.73	803.00 ± 2.00	855.00 ± 1.00	885.98 ± 0.05	971.33 ± 1.15

Note: Experimental data expressed as mean ± standard deviation (from triplicate measurement); A for 0.5 g, B for 1.0 g, C for 1.5 g, D for 2.0 g durian husk.

The behaviour and interactions throughout the adsorption process in terms of particle form and aggregation, and consequently the reactivity of organic molecules, could be an indication of the significance of adsorbent dose on pH. When contaminants are in contact with the durian husk (the adsorbent) during the sorption process, it makes for such contaminants to accumulate in aqueous environments [34]. The mechanism can either be classified as physisorption or chemisorption. The molecular interactions between the molecules of the adsorbate and the adsorbent are predominantly controlled by van der Waals forces during physical adsorption, as opposed to valence forces such as those involved in the production of chemical pollutants during chemisorption [35].

However, a previous study has reported that adsorption was efficient at neutral pH [36]. The physico-chemical changes of various materials used may affect the efficacy of the process. The area where a significant difference has been found between raw and NaOH-modified durian husk of 2.0 g adsorbent was the trend of pH value after 7 days. The pH of NaOH-modified durian husk is most likely approaching neutral. Therefore, 2.0 g of absorbent is recommended in this study.

3.2.2. Effect of Adsorbent Dose on Turbidity

As seen in Table 2, the turbidity of soaking water using raw and NaOH-modified increased gradually from the initial value until it reached the highest value on day 7 of soaking water. The initial turbidity of soaking water using 0.5 g, 1.0 g, 1.5 g, and 2.0 g of raw durian husk was 73.33 ± 1.53 NTU, 87.00 ± 1.00 NTU, 86.33 ± 0.58 NTU, and 86.39 ± 1.15 NTU, respectively. Then, the turbidity increased to 797.67 ± 0.58 NTU, 799.33 ±1.53 NTU, 873.52 ± 0.01 NTU, and 855.00 ± 1.73 NTU on day 7. Next, the turbidity for NaOH-modified durian husk also showed an increment from day 1 to day 7. On day 1, the values of turbidity were 93.00 ± 1.00 NTU, 102.33 ± 0.58 NTU, 114.00 ± 1.00 NTU, and 132.67 ± 2.08 NTU as the adsorbent dose increased from 0.5 g to 2.0 g. On the 7 days, the turbidity increased to 971.33 ± 1.15 NTU with 2.0 g of NaOH-modified adsorbent, as shown in Figure 3 (line graph).

Although the turbidity value increased in this investigation, the outcome was better than in earlier studies of the soaking water method. According to Megat Ahmad Azman et al. [5], the turbidity value on day 7 was 1103.30 ± 23.10 NTU. This study showed that the NaOH-modified durian husk helps to improve the soaking water quality for turbidity from 1103.30 ± 23.10 NTU to 971.33 ± 1.15 NTU. Overall, there was a slight difference observed in turbidity with treatment time. Soaking water during the retting process used raw black pepper rich in organic matter. Therefore, the soaking method produced a dark tan colour due to the de-husked berries during the retting process [37]. As time increased, the outer skin, pericarp, and outer surface of the mesocarp of well-matured berries were removed from the water. As a result, the soaking water became more acidic, and the dark colour leached into the water, and this is one of the variables influencing the turbidity value [38]. Thus, the wastewater cannot be discharged into the environment without proper treatment. This would be a fruitful area for further work to investigate the efficient method of wastewater treatment for pepper berries. More information on the wastewater treatment of pepper berries would help industries establish a greater quality of production in this matter.

3.2.3. Effect of Adsorbent Dose on COD

The result obtained for COD with raw and NaOH-modified durian husk is depicted in Table 3. The CODs for different dosages of 0.5 g, 1.0 g, 1.5 g, and 2.0 g raw durian husk at day 1 were 253.67 ± 3.21 mg/L, 255.00 ± 1.00 mg/L, 254.00 ± 1.00 mg/L, and 252.67 ± 1.53 mg/L, respectively. After day 7, the COD values increased to 1747.67 ± 26.58 mg/L, 1748.00 ± 1.00 mg/L, 1750.00 ± 19.47 mg/L, and 1760.67 ± 1.53 mg/L, as shown in Figure 4. The CODs serve as indicators of organic matter in the water. The presence of organic load contamination, as well as dissolved and suspended particles in the soaking water utilized in the retting process, clearly affects the COD value [39]. The COD value increases as the treatment time increases.

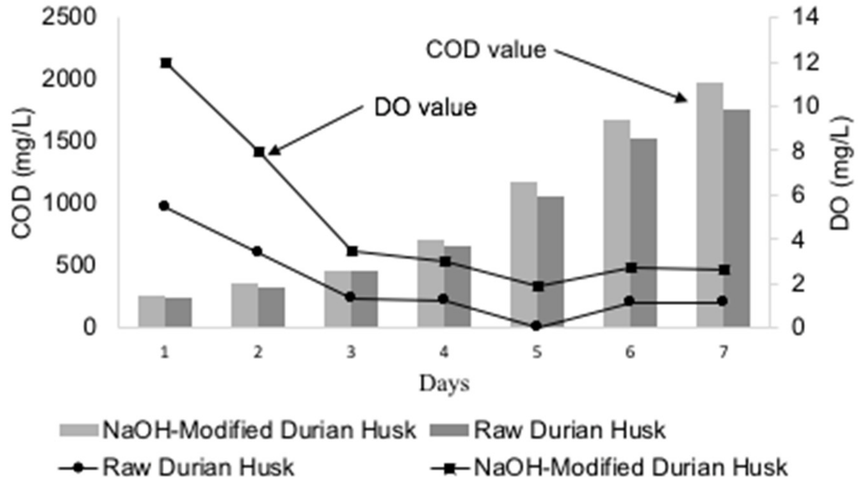

Figure 4. Effect of COD and DO for 2.0 g of raw and NaOH-modified durian husk.

Furthermore, Figure 4 shows an increment of COD value for NaOH-modified durian husk from day 1 to day 7. The values of COD on day 1 for 0.5 g, 1.0 g, 1.5 g, and 2.0 g were 250.00 ± 3.00 mg/L, 251.00 ± 1.73 mg/L, 256.33 ± 1.53 mg/L, and 265.33 ± 2.08 mg/L, increasing gradually to 1770.00 ± 17.32 mg/L, 1777.00 ± 1.73 mg/L, 1895.33 ± 1.53 mg/L, and 1984.67 ± 3.21 mg/L at day 7, respectively. In general, the value of COD of soaking water on day 7 with NaOH-modified durian husk has the highest value due to the crucial quantity of oxygen needed to decompose contaminants from suspended solids of pepper peel in the soaking water [40].

3.2.4. Effect of Adsorbent Dose on DO

Based on the data shown in Table 3, the DO of soaking water with 0.5 g of raw durian husk decreased sharply from 5.62 ± 0.01 mg/L on day 1 to 1.13 ± 0.01 mg/L on day 7, while the other conditions were from 5.42 ± 0.01 mg/L to 1.11 ± 0.01 mg/L, 5.41 ± 0.01 mg/L to 1.12 ± 0.09 mg/L, and 5.42 ± 0.01 to 1.13 ± 0.02 for 1.0 g, 1.5 g, and 2.0 g of adsorbent, respectively. The high reduction was observed from day 1 to 3 and decreased gradually on day 4 until the lowest on day 7. The DO value of NaOH-modified durian husk using 2.0 g of adsorbent also showed a reduction, as shown in Figure 4, but the lowest value of DO for modified durian husk was 1.49 ± 0.03 mg/L compared with raw durian husk, which was 1.13 ± 0.02 mg/L.

Based on previous work conducted by Megat Ahmad Azman et al. [6], the value of DO at day 7 was 6.15 ± 0.02 mg/L, which was higher than the DO value measured in this study. The DO value was high because of using flowing water instead of stagnant water. Water flow during the retting process may stop and replace oxygen lost during the decomposition of organic materials. DO value was decreased during the treatment time due to the long soaking period of pepper berries in water, where the bacteria naturally broke down the organic substance from the pepper berries, consuming some oxygen, which resulted in anaerobic conditions [41]. The suspended material from durian husk also may affect the DO in the water. As a result, soaking pepper berries in water will produce an odour.

3.3. Kinetic Study

Considering the promising chemical characterization results and potential of NaOH-modified durian husk as an adsorbent, the kinetic of soaking water for the different conditions of NaOH-modified durian husk was further assessed. The kinetic study is shown in Table 4.

Table 3. Mean value for COD and DO of soaking water with raw and NaOH-modified durian husk from day 1 to day 7.

Day	**COD (mg/L)**							
	Raw Durian Husk				NaOH-Modified Durian Husk			
	A	B	C	D	A	B	C	D
1	253.67 ± 3.21	255.00 ± 1.00	254.00 ± 1.00	252.67 ± 1.53	250.00 ± 3.00	251.00 ± 1.73	256.33 ± 1.53	265.33 ± 2.08
2	334.67 ± 2.52	335.00 ± 1.00	334.00 ± 2.00	335.00 ± 3.61	334.00 ± 2.94	335.00 ± 0.82	348.67 ± 3.06	357.00 ± 1.00
3	453.67 ± 4.73	457.33 ± 1.15	458.00 ± 1.00	464.67 ± 2.52	450.33 ± 1.53	451.00 ± 1.00	454.67 ± 1.53	463.33 ± 1.53
4	656.00 ± 1.00	659.00 ± 1.00	656.00 ± 1.00	664.00 ± 1.00	653.67 ± 1.53	697.00 ± 1.00	705.00 ± 1.00	714.00 ± 1.00
5	1061.00 ± 1.00	1058.33 ± 1.53	1067.00 ± 1.00	1068.67 ± 1.53	1056.33 ± 1.53	1157.00 ± 1.00	1166.67 ± 4.73	1170.33 ± 2.08
6	1462.67 ± 2.08	1476.00 ± 1.00	1466.00 ± 1.00	1523.33 ± 5.77	1457.00 ± 1.00	1486.00 ± 1.00	1496.33 ± 1.15	1672.33 ± 2.52
7	1747.67 ± 26.58	1748.00 ± 1.00	1750.00 ± 19.47	1760.67 ± 1.53	1770.00 ± 17.32	1777.00 ± 1.73	1895.33 ± 1.53	1984.67 ± 3.21
Day	DO (mg/L)							
	Raw Durian Husk				NaOH-Modified Durian Husk			
	A	B	C	D	A	B	C	D
1	5.62 ± 0.01	5.42 ± 0.01	5.41 ± 0.01	5.42 ± 0.01	5.69 ± 0.01	6.46 ± 0.02	6.48 ± 0.01	6.58 ± 0.02
2	3.45 ± 0.01	3.44 ± 0.01	3.42 ± 0.05	3.42 ± 0.02	3.57 ± 0.00	4.37 ± 0.00	4.47 ± 0.01	4.57 ± 0.08
3	1.32 ± 0.02	1.32 ± 0.02	1.31 ± 0.03	1.32 ± 0.02	1.41 ± 0.01	1.95 ± 0.02	1.97 ± 0.02	2.13 ± 0.02
4	1.27 ± 0.02	1.25 ± 0.01	1.25 ± 0.04	1.24 ± 0.01	1.30 ± 0.01	1.58 ± 0.02	1.67 ± 0.02	1.78 ± 0.01
5	1.24 ± 0.02	1.23 ± 0.01	1.21 ± 0.01	1..23 ± 0.01	1.26 ± 0.01	1.45 ± 0.01	1.65 ± 0.01	1.86 ± 0.02
6	1.18 ± 0.01	1.13 ± 0.02	1.12 ± 0.05	1.13 ± 0.01	1.25 ± 0.02	1.34 ± 0.01	1.42 ± 0.01	1.59 ± 0.01
7	1.13 ± 0.01	1.11 ± 0.01	1.12 ± 0.09	1.13 ± 0.02	1.20 ± 0.01	1.25 ± 0.01	1.34 ± 0.01	1.49 ± 0.03

Note: Experimental data expressed as mean ± standard deviation (from triplicate measurement); A for 0.5 g, B for 1.0 g, C for 1.5 g, D for 2.0 g durian husk.

Table 4. Kinetic of soaking water with raw and NaOH-modified durian husk.

Parameter	Condition	Zero-Order Model		First-Order Model		Second-Order Model	
		k	R^2	k	R^2	k	R^2
pH	A	−0.645	0.9706	−0.1118	0.9692	0.0228	0.9537
	B	−0.6589	0.9711	−0.1215	0.9732	0.0233	0.9415
	C	−0.6586	0.9689	−0.1217	0.9686	0.0234	0.933
	D	−0.6532	0.9646	−0.1207	0.9636	0.0233	0.927
Turbidity	A	117.91	0.9912	0.3691	0.9107	−0.0017	0.6829
	B	114.223	0.9605	0.3169	0.9686	0.0288	0.6463
	C	118.73	0.9726	0.3615	0.9465	0.0015	0.786
	D	121.17	0.9727	0.3657	0.9417	−0.0015	0.7797
DO	A	−0.6461	0.6493	−0.2507	0.713	0.1173	0.7683
	B	−0.63	0.6607	−0.2519	0.7254	0.1212	0.7853
	C	−0.6275	0.7209	−0.2513	0.7209	0.121	0.7776
	D	−0.6264	0.6561	−0.2496	0.7174	0.1194	0.7718
COD	A	262.33	0.9439	0.3425	0.9905	−0.0006	0.9384
	B	262.93	0.9436	0.3421	0.9905	−0.0005	0.9384
	C	262.89	0.9444	0.3426	0.9904	−0.0016	0.9375
	D	268.02	0.9427	0.3459	0.9898	−0.00083	0.9844

Note: Experimental data expressed as A for 0.5 g, B for 1.0 g, C for 1.5 g, D for 2.0 g durian husk; k, rate constant (day^{-1}); R^2, coefficient.

For pH value, the R^2 values for zero-, first-, and second-order kinetic models of condition A were 0.9706, 0.9692, and 0.9537, while B were 0.9711, 0.9732, and 0.9415, respectively. On the other hand, the R^2 values for condition C were 0.9689, 0.9686, and 0.933, while condition D showed the R^2 values of 0.9646, 0.9636, and 0.927 for zero-, first-, and second-order kinetic models, respectively. In addition, the rate constant for pH increased from −0.645, −0.6589, −0.6586, to −0.6532 (day^{-1}) and increased steadily until 0.0228, 0.0233, 0.0234, and 0.0233 (day^{-1}) for conditions A, B, C, and D, respectively. Overall, condition B had the highest R^2 value agreement with the first-order kinetic model. The R^2 around 1 indicated that the model was completely fit; however, an R^2 near 0 indicated that the model could not be employed due to substantial variance differences [42].

The turbidity values for condition A were 0.9912, 0.9107, and 0.6829 for zero-, first-, and second-order kinetic models, respectively. Condition B's values were 0.9711, 0.9686, and 0.6463, while the R^2 values for condition C were 0.9726, 0.9417, and 0.7797 for zero-, first-, and second-order kinetic models, respectively. In contrast, the R^2 values of 0.9727, 0.9417, and 0.7797 were obtained for zero-, first- and second-order kinetic models for condition D. What can be clearly seen in the rate constant for turbidity is the variability of trend. The peculiar trend may be attributed to the complex adsorbent–adsorbate interactions between pepper berry solution and different adsorbent doses [43].

The R^2 values of COD for condition A were 0.9439, 0.9905, and 0.9384 for zero-order, first-order, and second-order kinetic models, respectively. For COD in condition B, the R^2 of the zero-, first-, and second-order kinetic models were 0.9436, 0.9905, and 0.9384, respectively. Furthermore, the R^2 values obtained for condition C were 0.9444, 0.9904, and 0.9375 for the zero-order, first-order, and second-order kinetic models, respectively. In contrast, the R^2 of 0.9427, 0.9898, and 0.9844 were reported for zero-, first-, and second-order kinetic models (condition D). Similar peculiar findings were also revealed for the rate constant of COD. The rate constant of COD for the zero-order kinetic models increased from 262.33, 262.93, 262.89, to 268.02 day^{-1} for conditions A, B, C, and D, respectively. However, the rate constant of COD for the first order exhibited a descending trend from 0.3425 to 0.3421, ascending from 0.3426 to 0.3459 day^{-1} for conditions A, B, C, and D, respectively. Second-order kinetic model analysis showed the rate constant of COD increased from −0.0006 to −0.0005 for conditions A and B, respectively. A further reduction of −0.0016 and an increment of −0.00083 were observed for conditions C and D, respectively.

DO also indicated that the R^2 values obtained in condition A were 0.6493, 0.6607, 0.7209, and 0.6561 (zero-order model); 0.713, 0.7254, 0.7209, and 0.7174 (first-order model); and 0.7683, 0.7853, 0.7776, and 0.7718 (second-order model). Thus, the R^2 value for DO fits with the second-order kinetic model based on the higher value obtained compared to others. In addition, the rate constant for DO was increased respective to time from −0.6461, −0.6300,−0.6275, and −0.6264 day^{-1} to −0.2507, −0.2519, −0.2513, and −0.2496 day^{-1} until 0.1173, 0.1212, 0.1210, and 0.1194 $days^{-1}$ for conditions A, B, C and D.

Overall, there is a relatively small body of literature that is concerned with kinetic study of soaking water for pepper berries. Megat Ahmad Azman et al. [5] investigated different kinetic models for different amounts of gunny bags of pepper berries. They found that water quality changes in turbidity, pH, DO, and COD during the retting process were adequately explained by zero-, first- and second-order kinetic models. The R^2 values show promising results for all parameters studied for the zero-order model except for DO analysis. Nevertheless, the results obtained from this study are somewhat counterintuitive. Comparing the different kinetic models, the first-order model reveals the most significant R^2 values for pH, turbidity, and COD analysis except for DO analysis. The discrepancies or inconsistencies of results may be due to the errors and misconceptions in the approaches deployed to linearizing the adsorption models [44]. Another possible reason may be the introduction of adsorbents and their modifications that alter the physico-chemical nature of water quality parameters [45]. Furthermore, a greater focus on analysis of the components that are generated in the water that may or may not be adsorbed by the adsorbents, including energy-dispersive X-ray spectroscopy and pH point of zero charge, could be conducted in the future, and these results could produce interesting findings that account more for physisorption or chemisorption properties.

Additionally, complicated interactions that occur between the solute and solid during the retting process due to modified adsorbents may have affected the outcomes. Despite the limitations of the understanding of the surface heterogeneity of the material, the findings do however suggest that future research could focus on either of the Freundlich or Langmuir adsorption isotherm models to describe the adsorption processes of the adsorbents [22]. The relationship of equilibrium concentration of substrate at a constant temperature and the amount of a material removed from the liquid phase by unit mass of adsorbent could be explained by the adsorption isotherm [46].

4. Conclusions

This study revealed the potential of durian husk as an efficient adsorbent to improve the soaking water of the retting process of pepper berries. The well-developed porous structure, with a BET surface area of 1.51 m^2/g (raw durian husk) and 2.33 m^2/g (NaOH-modified durian husk), respectively, enhance the adsorption process. The results obtained from this study show that the raw and NaOH-modified durian husk improved the water quality of the soaking water. All the parameters highly influence the water properties. The effects of adsorbent on pH, turbidity, COD, and DO was observed. The 2.0 g of NaOH-modified durian husk enhanced the greatest changes of the four parameters. The highest pH value using NaOH-modified durian husk was 6.10 ± 0.02, while turbidity and COD increased to 971.33 ± 1.15 NTU and 1984.67 ± 3.21 mg/L, respectively. In addition, the DO of NaOH-modified durian husk showed the highest reduction to 1.49 ± 0.03 mg/L. Equilibrium studies showed that the first-order kinetic model fit the adsorption data for 2.0 g NaOH-modified durian husk. Considerably more work will need to be performed to clarify the results by various modern techniques, and it will also be necessary to look at the adsorption mechanism. Further study is also required to observe the turbidity value and DO of wastewater. The findings of the reduction of wastewater turbidity or certain thresholds of DO have a number of important implications for future practice.

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