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Synthesis and Characterization of Natural Pahae Zeolite–Durian Peel Activated Carbon as an Adsorbent for Phosphate Reduction in Laundry Wastewater

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ABSTRACT

This study aims to synthesize and characterize composites based on Pahae Natural Zeolite and Durian Peel Activated Carbon as phosphate reducers in laundry wastewater. Zeolite was activated using 1 M NaCl solution, while activated carbon was prepared by carbonization at 500 °C for 2 hours. The Zeolite–Activated Carbon mixtures were prepared with compositions of 100%:0%, 95%:5%, 90%:10%, 85%:15%, 80%:20%, 75%:25%, and 0%:100%, pressed at 6 tons for 10 minutes, and sintered at 700 °C, 800 °C, and 900 °C for 4 hours. Characterization included physical properties (porosity, water absorption), mechanical properties (hardness), morphology (SEM), crystal structure (XRD), and elemental composition (XRF). The results showed that the optimum condition was achieved at a 75%:25% composition and 700 °C, with porosity of 59.67%, water absorption of 56.06%, and phosphate reduction efficiency of 80.86%. The highest hardness value was obtained at 100%:0% composition at 900 °C, reaching 784.14 MPa. Therefore, the Pahae Natural Zeolite–Durian Peel Activated Carbon composite demonstrates potential as an effective and environmentally friendly adsorbent for wastewater treatment.

Keywords: Adsorption, Durian Peel Activated Carbon, Laundry Wastewater, Natural Pahae Zeolite, Phosphate

ABSTRAK

Penelitian ini bertujuan untuk mensintesis dan mengkarakterisasi komposit berbasis Zeolit Alam Pahae dan Arang Aktif Kulit Durian sebagai pereduksi fosfat pada limbah laundry. Zeolit diaktivasi menggunakan NaCl 1 M, sedangkan arang aktif diperoleh melalui karbonisasi pada 500 °C selama 2 jam. Campuran Zeolit–Arang Aktif divariasikan dengan komposisi 100%:0%, 95%:5%, 90%:10%, 85%:15%, 80%:20%, 75%:25%, dan 0%:100%, kemudian dicetak pada tekanan 6 ton selama 10 menit dan disinter pada suhu 700 °C, 800 °C, dan 900 °C selama 4 jam. Karakterisasi meliputi sifat fisis (porositas, daya serap air), mekanik (kekerasan), morfologi (SEM), struktur kristal (XRD), serta kandungan unsur (XRF). Hasil penelitian menunjukkan kondisi optimum pada komposisi 75%:25% dengan suhu 700 °C, menghasilkan porositas 59,67%, daya serap air 56,06%, dan reduksi fosfat sebesar 80,86%. Nilai kekerasan tertinggi diperoleh pada komposisi 100%:0% pada suhu 900 °C sebesar 784,14 MPa. Dengan demikian, komposit Zeolit Alam Pahae–Arang Aktif Kulit Durian berpotensi sebagai adsorben efektif dan ramah lingkungan untuk pengolahan limbah cair.

Kata kunci: Adsorpsi, Arang Aktif Kulit Durian, Fosfat, Limbah Laundry, Zeolit Alam Pahae

1. Introduction

Water pollution is one of the serious environmental issues that significantly affects public health and ecosystems. One major source of water pollution is laundry wastewater, which generally contains harmful chemicals such as phosphates, surfactants, ammonia, and nitrogen. Laundry detergents typically contain 70–80% phosphate and about 20–30% surfactants [1]. According to the Regulation of the Minister of Environment of the Republic of Indonesia No. 5 of 2014, the maximum permissible phosphate concentration in laundry wastewater is 2 mg/L [2]. Therefore, effective wastewater treatment technologies are urgently needed to reduce phosphate levels before being discharged into the environment.

Among various treatment methods, adsorption has been widely applied by utilizing materials such as zeolite, activated carbon, and silica sand [3]. Natural zeolite is known for its porous structure, large surface area, and excellent ion-exchange properties, making it highly effective as an adsorbent [4]. Zeolite was first discovered by Baron Axel Frederick Cronstedt in 1756, and to date, it has been found in various locations both on land and at the seabed [5].

In addition to zeolite, activated carbon is also commonly used as an adsorbent. In this study, activated carbon is produced from durian peel waste. Durian peel is chosen because it is an abundant agricultural byproduct, accounting for about 57–75% of the total fruit weight, and may cause environmental problems if not utilized properly [6]. Durian peel contains a high carbon content, which allows it to be converted into activated carbon through carbonization and activation processes [7].

Previous studies have demonstrated the effectiveness of combining zeolite and activated carbon in reducing water pollutants. For instance, natural Yemeni zeolite modified with 1 M NaCl at 80 °C achieved a phosphate removal efficiency of 98.60% [8]. Another study reported that zeolite with particle sizes ranging from 50–120 mesh, activated using HCl and Mg^{2+}, achieved phosphate adsorption efficiency up to 85.30% [9]. Research using natural Pahae zeolite combined with cocoa shell-based activated carbon successfully reduced ammonium concentration in greywater by 75.95% [10]. Furthermore, activated carbon derived from durian peel carbonized at 500 °C for 2 hours with a particle size of 200 mesh was found to meet the Indonesian National Standard (SNI 06-3730-1995), with a moisture content of 14.12%, ash content of 5.46%, and iodine adsorption capacity of 580.27 mg/g [11].

Based on this background, the present study focuses on the synthesis and characterization of Natural Pahae Zeolite – Durian Peel Activated Carbon as an adsorbent material for reducing phosphate content in laundry wastewater. This research is expected to provide an alternative approach for the utilization of durian peel waste while offering an environmentally friendly solution for domestic wastewater treatment.

2. Method

2.1. Materials

The main materials used were natural Pahae zeolite (North Tapanuli, North Sumatra) and durian peel (Durio zibethinus L.) obtained from local vendors in Medan. Supporting chemicals included NaCl (Merck), distilled water, and standard reagents for phosphate analysis.

2.2. Preparation of Natural Pahae Zeolite

The zeolite was crushed, sieved (200 mesh, 75 µm), washed with distilled water, and then dried at 100 °C for 24 hours. Activation was carried out using 1 M NaCl solution at 80 °C with stirring at 120 rpm for 1 hour, followed by drying again at 70 °C for 24 hours.

2.3. Preparation of Durian Peel Activated Carbon

The durian peel was cleaned, cut into small pieces, and carbonized at 500 °C for 2 hours. The carbonized product was then ground, sieved (200 mesh), and characterized according to the Indonesian National Standard (SNI 06-3730-1995), which includes fixed carbon content, ash content, moisture content, and volatile matter.

2.4. Preparation of Zeolite–Durian Peel Activated Carbon Composites

Zeolite and durian peel activated carbon were mixed with composition variations of 0%:100% (A1), 100%:0% (A2), 95%:5% (A3), 90%:10% (A4), 85%:15% (A5), 80%:20% (A6), and 75%:25% (A7). The mixtures were molded into block shapes (10 g, dimensions 1×3×3 cm) using a hydraulic press (6 tons, 10 minutes), then dried and activated at 700 °C, 800 °C, and 900 °C for 4 hours.

2.5. Characterization of Composites

Characterization was conducted to determine the physical properties (porosity, water absorption), mechanical properties (Vickers hardness test), elemental and compound content using XRF, as well as crystal structure and morphology analysis using XRD and SEM.

2.6. Phosphate Adsorption Test

The initial phosphate concentration of laundry wastewater was determined using a UV-Vis spectrophotometer in accordance with SNI 6989-31:2021. The adsorption process was carried out by mixing 10 g of composite into 100 mL of laundry wastewater at 25 °C, stirred with a shaking water bath (135 rpm, 1 hour). The mixture was then filtered, and the remaining phosphate concentration was re-analyzed to calculate the removal efficiency.

2.7. Porosity Test

Porosity values were determined based on ASTM C-373-88-2006 standards. Porosity was calculated as the percentage of pore volume filled with water relative to the total sample volume after firing. The calculation used wet mass (m_b), dry mass (m_k), water density (ρ_{water}), and sample volume after firing (V_t), according to Equation (1):

$$\%\text{porosity} = \left(\frac{m_b - m_k}{\rho_{water} \times V_t}\right) \times 100\% \quad (1)$$

2.8. Water Absorption Test

Water absorption was determined based on ASTM C-20-00-2005 standards. The samples were immersed in water at room temperature for 24 hours, then weighed before and after immersion. The water absorption value was calculated from the difference between wet mass (m_b) and dry mass (m_k) relative to the dry mass, as shown in Equation (2):

$$\text{Water Absorption} = \left(\frac{m_b - m_k}{m_k}\right) \times 100\% \quad (2)$$

2.9. Hardness Test (Vickers Hardness)

The hardness test was carried out using a Vickers Hardness tester on samples that had been physically activated at temperatures of 700 °C, 800 °C, and 900 °C. The hardness value was obtained by applying a specific load (F) to the sample surface and then measuring the diagonal length of the resulting indentation (d). The Vickers Hardness (H_v) was calculated using Equation (3):

$$\text{H}_\text{v} = 1.8544 \frac{F}{d^2} \quad (3)$$

3. Result and Discussion

3.1. Porosity Analysis

Figure 1 presents the results of porosity testing on samples with different compositions and sintering temperatures. The highest porosity value of 59.67% was obtained in sample A7 (composition 75%:25% at 700 °C). Conversely, the lowest porosity value of 34.40% was found in sample A2 (composition 100%:0% at 900 °C). These results indicate that the higher the sintering temperature, the lower the porosity value tends to be. This phenomenon is attributed to the more intensive sintering process at elevated temperatures, which causes the pores to become denser and the pore size to decrease. In addition, the results also demonstrate that the addition of activated carbon filler from durian peel significantly increases porosity. This enhancement occurs because activated carbon possesses a porous structure that can enlarge both the number and size of pores in the adsorbent material.

These findings are consistent with a previous study [12], which reported that filters based on Pahae Natural Zeolite–Cocoa Peel Activated Carbon with a 75%:25% composition at 700 °C exhibited a porosity value of 53.01%. In contrast, filters with a 100%:0% composition showed a lower porosity value of 23.80%. Thus, it can be concluded that the combination of Pahae Natural Zeolite and durian peel activated carbon at certain compositions is capable of producing adsorbent materials with improved porosity characteristics, particularly at relatively low firing temperatures (700 °C).

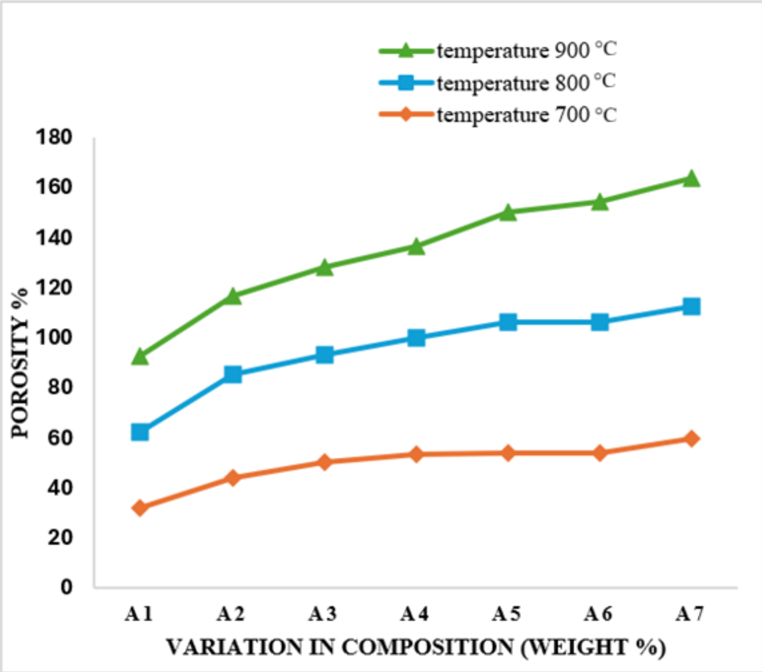

Figure 1. Graph of porosity test results.

3.2. Water Absorption Analysis

Figure 2 shows the water absorption results for various compositions and sintering temperatures. The highest water absorption value was obtained in sample A7 (composition 75%:25%, sintering temperature 700 °C), reaching 56.06%. Conversely, the lowest value of 32.74% was observed in sample A2 (composition 100%:0%, sintering temperature 900 °C).

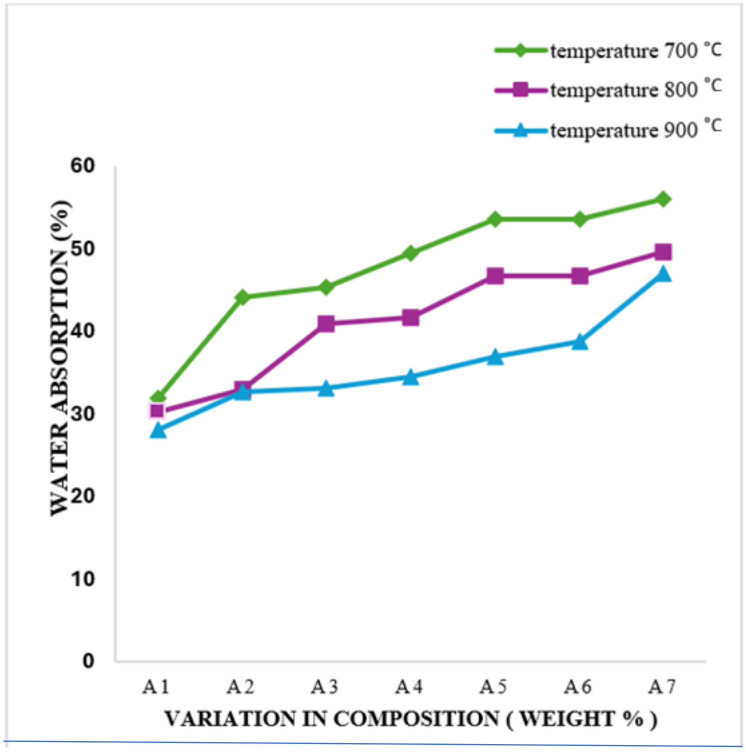

Figure 2. Graph of water absorption test results.

In general, Pahae Natural Zeolite has an aluminosilicate framework with uniform cavities that act as sites for molecular adsorption. However, its adsorption capacity is limited by the pore size of the zeolite. Meanwhile, activated carbon derived from durian peel is dominated by amorphous carbon, which possesses a high internal surface area and a wide distribution of micro–mesopores, thereby providing greater adsorption capability. Therefore, the addition of durian peel activated carbon filler was proven to enhance the water absorption capacity of the adsorbent material.

These results also indicate that water absorption is directly proportional to porosity. The greater the fraction of activated carbon added, the more open the pore structure becomes, allowing water to be more readily adsorbed. Conversely, an increase in firing temperature reduces water absorption, as the sintering process causes the pores to become denser and partially closed.

This finding is consistent with a previous study [12], which reported that an adsorbent based on Pahae Natural Zeolite–Cocoa Peel Activated Carbon achieved optimal water absorption at a 75%:25% composition at 700 °C, with a value of 53.01%, whereas the 100%:0% composition at 900 °C reached only 29.75%. Thus, the combination of zeolite and durian peel activated carbon with a balanced composition and an appropriate sintering temperature (700 °C) provides the most optimal water adsorption characteristics.

3.3. Hardness Analysis

Figure 3 presents the hardness test results of the adsorbent based on Pahae Natural Zeolite–Durian Peel Activated Carbon. The highest hardness value was obtained in sample A1 (composition 100%:0%, sintering temperature 900 °C), reaching 784.14 MPa. In general, increasing the sintering temperature contributed to higher hardness values. This is attributed to the densification process at elevated temperatures, which reduces pore diameter and produces a more compact material structure. Conversely, the addition of durian peel activated carbon filler tended to decrease the hardness of the adsorbent. This phenomenon indicates that hardness is inversely related to porosity and water absorption. As the fraction of activated carbon increases, the adsorbent structure indeed develops more pores that enhance adsorption properties, but this simultaneously reduces density and mechanical strength.

Compared with a previous study [12], the hardness value for the 100%:0% composition at 900 °C was reported as 351.31 MPa. The present study shows a similar trend, although the hardness values obtained here are relatively higher. This suggests that the applied activation method and sintering conditions were effective in improving the mechanical properties of the material, although the optimum hardness value has not yet been fully achieved.

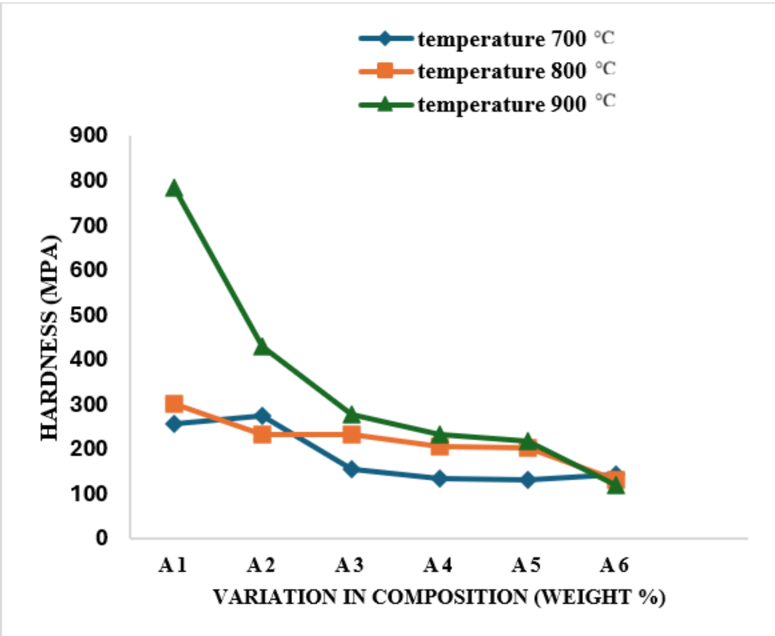

Figure 3. Graph of composite hardness test results.

3.4. SEM Morphological Analysis

Morphologically, the surface of samples with filler addition exhibited a rougher texture, wider cavities, and higher pore connectivity. This phenomenon is likely due to the intrinsic characteristics of activated carbon, which possesses a porous structure with micro- and mesopores. When incorporated into the matrix, it increases the number of pore sites.

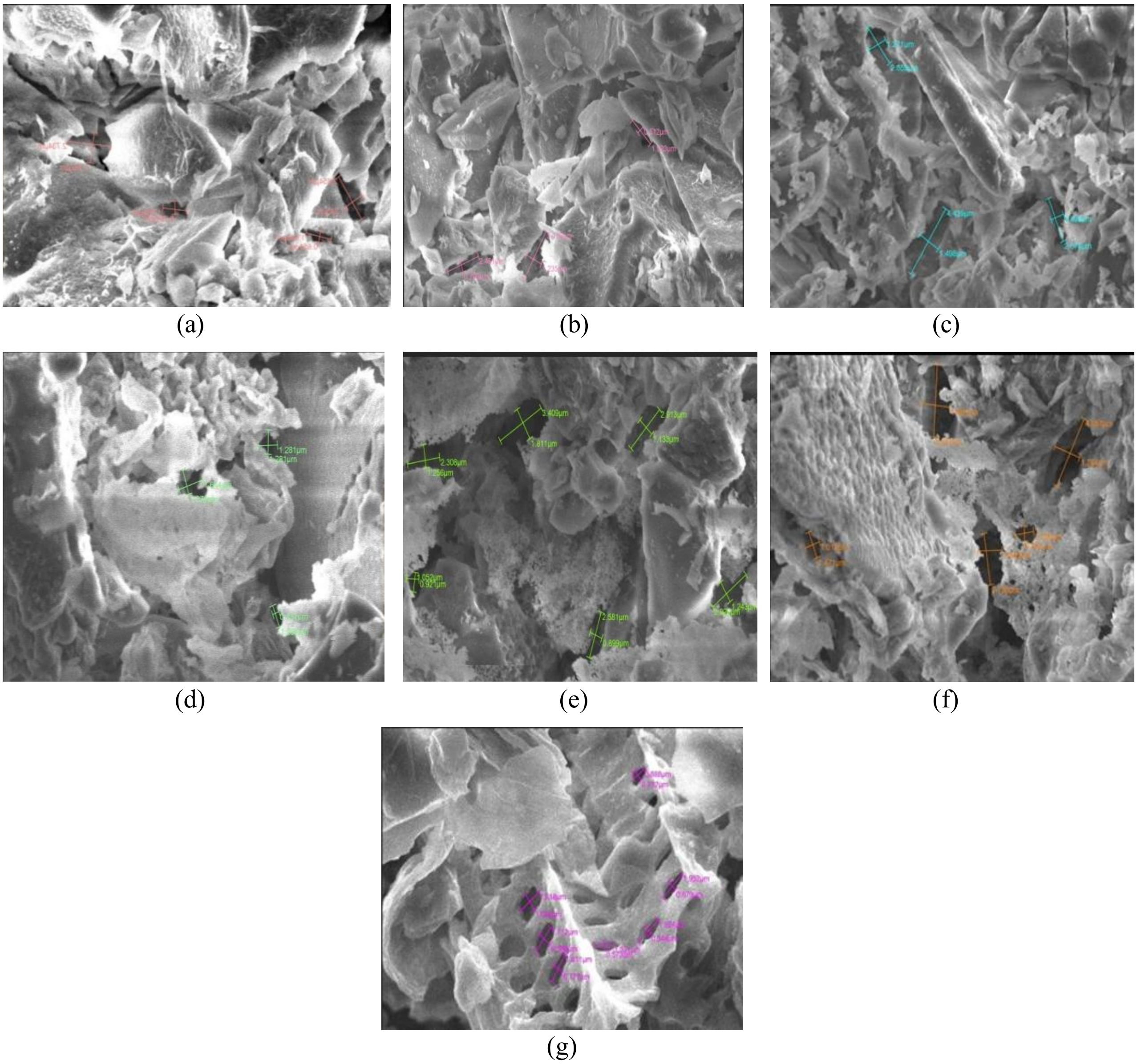

Figure 4**.** SEM results at 5000× magnification and 700 °C with composition variations: (a) 100%:0%, (b) 95%:5%, (c) 90%:10%, (d) 85%:15%, (e) 80%:20%, (f) 75%:25%, (g) 0%:100%.

These findings are consistent with previous reports [13], which state that the incorporation of activated carbon materials into composites can enlarge pore size and increase specific surface area. The implication of these results is that a higher content of durian peel activated carbon filler enhances the total porosity of the samples. This condition potentially improves the adsorption performance of the material by enlarging the contact area between the adsorbent and the fluid passing through the pores. However, it should be noted that excessive increases in porosity and pore size may also affect the mechanical properties of the material, such as reducing compressive strength due to lower structural density. Thus, the addition of durian peel activated carbon filler has been proven to significantly influence the increase in porosity and pore size of the material, thereby supporting its potential for filtration and adsorption applications.

3.5. XRF Analysis

Table 1 shows the XRF analysis results of Pahae Natural Zeolite activated with NaCl. The main elements present are Silica (Si) at 39.027% and Aluminum (Al) at 7.849%, with a total Si–Al content of 46.876%. The Si/Al ratio is 4.972; therefore, based on the classification [14], this zeolite falls into the category of medium-silica zeolite and can be classified as modernite-type zeolite (Si/Al molar ratio = 2–5). In addition, the zeolite also contains other significant elements such as K (18.465%), Fe (13.722%), and Ca (13.05%).

Table 2 shows the elemental composition of the durian peel activated carbon filler. The dominant element is Carbon (C) at 70.512%, followed by Calcium (Ca) at 21.465%. Other elements such as P, S, Si, Fe, and Cu are present in smaller amounts. The high carbon content makes activated carbon a promising adsorbent material, while impurities such as P, Fe, Cu, and S may cause pore blockage, thereby reducing the active surface area [15].

Table 1. Elemental composition of Pahae Natural Zeolite activated with NaCl.

Element	Concentration (%)
Si	39.027
K	18.465
Fe	13.722
Ca	13.05
Al	7.849
P	3.105
Mn	0.155

Table 2. Elemental composition of durian peel activated carbon filler.

Element	Concentration (%)
C	70.512
Ca	21.465
P	2.867
S	1.143
Si	0.503
Fe	0.268
Cu	0.112

3.6. XRD Analysis

Figure 5 shows the XRD test results for variations in the adsorber composition. At the 100%:0% composition (pure zeolite) at 700 °C, diffraction peaks appear at 2θ = 24°–30°, indicating the presence of the modernite zeolite phase. In general, the crystal peaks of all compositions appear broad, except for the 0%:100% composition (durian peel activated carbon), which shows sharper peaks at around 2θ ≈ 30°. This is consistent with the characteristics of activated carbon, which tends to be amorphous and thus does not significantly increase crystal intensity [16].

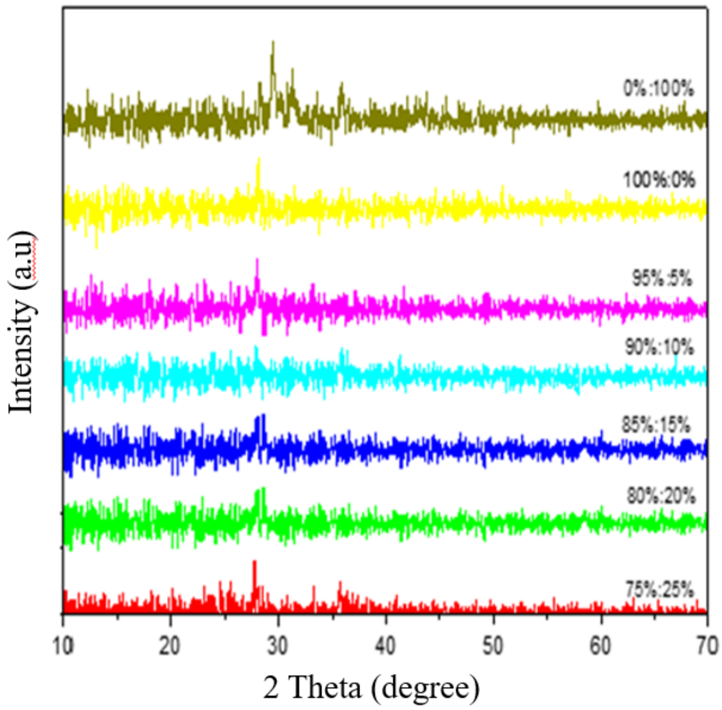

Figure 5. XRD test results.

Previous studies reported that Pahae Natural Zeolite with a particle size of 325 mesh that had been activated also exhibited the mordenite phase at 2θ = 25°–30° [17]. Thus, the XRD diffraction pattern in this study confirms that the Zeolite–Activated Carbon mixture is dominated by amorphous characteristics, which are expected to enhance the adsorptive properties of the material.

3.7. Phosphate Adsorption Analysis in Laundry Wastewater

The results of the physical property tests showed that the optimum composition was found in the 75%:25% mixture (Pahae Natural Zeolite : Durian Peel Activated Carbon) at a temperature of 700 °C. Therefore, this composition was used for the adsorption test of phosphate content in laundry wastewater.

Table 3. Results of application testing on laundry wastewater with a contact time of 1 hour.

Phosphate Content (mg/L)		Percentage Reduction of Phosphate Content (%)
Before	After	
2.15648	0.41272	80.86

Based on Table 3, the phosphate content in laundry wastewater decreased from 2.15648 mg/L to 0.41272 mg/L after the adsorption process for 1 hour. The percentage reduction reached 80.86%, indicating the effectiveness of the Pahae Natural Zeolite–Durian Peel Activated Carbon-based adsorber in reducing phosphate content. When compared with previous research [18], which successfully reduced phosphate up to 98.60%, this result is still below the optimum performance. However, in comparison with the findings of Wirosoedarmo et al. (2018), who reported phosphate reduction percentages ranging from 47.56% to 71.93% in laundry wastewater, the 80.86% reduction achieved in this study can be considered superior [19].

Furthermore, previous research by Susilawati et al. (2023), demonstrated that a Pahae Natural Zeolite–Cocoa Peel Activated Carbon-based filter was able to reduce ammonium by 75.56% at a 75%:25% composition at 700 °C [20]. Compared to that, the Pahae Natural Zeolite–Durian Peel Activated Carbon adsorber under the same conditions yielded higher performance in phosphate removal, achieving a reduction of 80.86%.

In conclusion, the combination of Pahae Natural Zeolite–Durian Peel Activated Carbon at a 75%:25% composition and a sintering temperature of 700 °C proved effective in reducing phosphate content in laundry wastewater, although there is still room for optimization to approach the nearly perfect efficiency reported in previous studies.

4. Conclusion

An adsorber based on Pahae Natural Zeolite–Durian Peel Activated Carbon was successfully fabricated and applied as a phosphate-reducing material for laundry wastewater. Characterization results showed that the optimum condition was achieved at a 75%:25% composition and a sintering temperature of 700 °C, yielding a porosity of 59.67% and a water absorption capacity of 56.06%. The highest hardness value was obtained at a 100%:0% composition and 900 °C, reaching 784.14 MPa. The addition of durian peel activated carbon was found to increase porosity and water absorption capacity, but decrease the material's hardness. Conversely, higher sintering temperatures improved hardness but reduced porosity and water absorption. In laundry wastewater application, the adsorber under optimum conditions successfully reduced phosphate concentration from 2.15648 mg/L to 0.41272 mg/L, equivalent to a removal efficiency of 80.86% within 1 hour. Therefore, this material has strong potential to be used as an environmentally friendly filtration medium for wastewater treatment.

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