Immobilization of activated carbon in fractionated clay from East Kalimantan as wastewater adsorption material

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Abstract. The aim of this research is to make immobilization of activated carbon in fractionated clay from East Kalimantan as wastewater adsorption material. Fractionated clay and activated carbon were made from the lai shell, then the adsorbents were immobilized using a ratio of 2.5:5 (g activated carbon: g clay) to increase their adsorption ability to wastewater. Furthermore, the adsorbents were characterized using X-Ray Diffraction (XRD) to analyze the phase, composition, and crystal structure and Scanning electron microscopy (SEM) to analyze the morphology of adsorbent and Fourier Transform Infrared (FTIR) to analyze the functional group. The results of adsorbent characterization by XRD showed that the sample contains 70% SiO₂ phase which is shown at the highest and sharpest peak, namely at 26.47°. The results of SEM analysis on immobilized adsorbents indicates the presence of silica nanoparticles that appear as white aggregates, where this silica is a compound contained in the clay. Whereas the results of the adsorbent characterization using FTIR showed that the functional groups in fractionated clay were Si-OH (silanol), Al-O-Al, Si-C and Si-O-Si (siloxane). In activated carbon found several functional groups namely C-H (alkyne), C=C (aromatics) and alkylhalide. Whereas the immobilized adsorbents with a ratio of 2.5:5 are Si-OH, Si-O-Si, Al-O-Al, alkylhalide and C-H. This shows that immobilized adsorbents have formed quite well.

1. Introduction
Industrial development is one source of environmental pollution problems through industrial waste containing heavy metals. Most of these heavy metals can pollute waters in the form of heavy metal ions. This heavy metal is dangerous because it cannot be degraded in the body, but accumulates over a certain period of time. This can have toxic effects on living things even if only in low concentrations. These heavy metal ions can be reduced by adsorption methods using activated carbon and clay. Activated carbon is used because it has pores that are very effective in absorbing or binding heavy metal ions from solution. Activated carbon material obtained from the fruit skin typical of the Kalimantan region is lai (Durio kutejensis (Hassk) Becc.). This fruit is still a family with durian fruit (Durio zibethimus), so both of these plants have almost the same skin composition [1]. Durian skin composition consists of cellulose elements of 50-60%, 5% lignin, and 5% starch. High carbon content is also found in durian skin, so this material can be processed into activated carbon adsorbents [2]. Natural clay adsorbents are used because they
have active sites in the form of silanol (Si-OH), aluminol (Al-OH), siloxane (Si-O-Si) and iron oxide which can adsorb metal ions effectively. Both of these adsorbents can also be increased the ability of adsorption with the immobilization method. So, in this research the synthesis of activated carbon from the lai shell (*Durio kutejensis* (Hassk) Becc.) and fractionating clay, which is then carried out an immobilization system between the adsorbents to increase the ability of adsorption against wastewater.

2. Experimental method

2.1. Materials

Fruit skin of lai (*Durio kutejensis* (Hassk) Becc.), Clay, HCl, KMnO₄, H₂SO₄, Aquades. All the reagents were used as received without further purification.

2.2. Synthesis of Activated Carbon

Lai shell with a size of 1x2 cm² is dried and carbonized using a furnace at 400°C for ± 15 minutes. Then the charcoal is cooled and ground and then sieved with a 100 mesh sieve. Then activated using HCl solution for 24 hours. Then filtered and washed. After that, it is dried at 100°C for 3 hours. Then 1 g of activated carbon is cooled in a desiccator to calculate its water content [3].

2.3. Synthesis of Fractionated Clay

Dry clay was sieved using a 100 mesh sieve and mixed with KMnO₄ while stirring for 4 hours at 80°C. The results are filtered and washed and then roasted for 12 hours at 80°C. Then this clay was added H₂SO₄ with the same procedure and also HCl with the same procedure.

2.4. Immobilization of Lai *Durio kutejensis* (Hassk) Becc. in Clay

Activated carbon from lai shell is mixed with clay using a ratio of 2.5: 5 (g activated carbon: g clay).

2.5. Characterization

Scanning electron microscopy (SEM) was applied to analyze the morphology of adsorbent. X-Ray Diffraction (XRD) was applied to analyze the phase, composition, and crystal structure. Fourier Transform Infrared (FTIR) was applied to analyze the functional group.

3. Results and Discussion

3.1. X-Ray Diffraction (XRD) analysis

XRD testing is carried out to determine the phase structure formed in a sample. The samples used in this test were 0.25 grams of activated carbon from the peel of lai fruit and 0.5 grams of fractionated clay which were immobilized. The results of XRD analysis of the immobilized adsorbent with a particle size of 200 mesh are shown in Figure 1. Based on Figure 1, it can be seen that the sample contains 70% SiO₂ phase which is shown at the highest and sharpest peak, namely at 26.47°, the low quartz phase is 15.6% and zeolite 14.4%. This shows that the phases formed are mostly from fractionated clay samples.
3.2. Scanning electron microscopy (SEM) analysis

The results of surface morphology scans of immobilized activated carbon in clay can be seen in Figure 2, where SEM sample testing is carried out at magnifications of 10 to 30 µm.

![Figure 1. XRD spectrum of immobilized adsorbents with ratio 25:5](image1)

**Figure 1.** XRD spectrum of immobilized adsorbents with ratio 25:5

![Figure 2. SEM of immobilized adsorbents with ratio 25:5](image2)

**Figure 2.** SEM of immobilized adsorbents with ratio 25:5: (a) 50 µm magnification, (b) 30 µm magnification, (c) 10 µm magnification
Figure 2a with 50 μm magnification shows the morphological form of immobilized adsorbents which is not uniform in shape or size. In Figure 2b with 30 μm magnification, it can be seen that the most dominant form is sheet-shaped and a small portion of the particles are in the form of granules and there is porosity, in this picture you can also see activated carbon which is dark black. Meanwhile, the 10 μm magnification shown in Figure 2c indicates the presence of silica nanoparticles that appear as white aggregates [10], where this silica is a compound contained in the clay. So that from the results of this SEM test, it can be seen that the immobilized adsorbent sample has been formed well.

3.3. Fourier Transform Infrared (FTIR) analysis

The FTIR spectrum for samples of activated carbon, fractionated clays and immobilized adsorbents is shown in Figure 3 below.

![Figure 3. FTIR spectrum: (a) fractionated clay, (b) immobilized adsorbents, (c) activated carbon](image)

Based on Figure 3, spectrum a shows that the O-H group on Si-OH (silanol) appears at the wavelength 3619 – 3695 cm\(^{-1}\) which indicates the presence of water absorbed in the clay. Si-OH groups appear at a wavelength of 1637 cm\(^{-1}\), while Si-O-Si (siloxane) groups appear at wavelengths of 1003, 527 and 424 cm\(^{-1}\). In addition, the Al-O-Al group is shown at a wavelength of 795 and 776 cm\(^{-1}\). And at a wavelength of 692 cm\(^{-1}\), a Si-C group appears. Spectrum b indicates that the absorption band is not much different from spectrum a, because the sample in spectrum B is the result of immobilization between activated carbon and
clay. The O-H group on silanol remains at 3619 – 3695 cm⁻¹, then there is an additional group at a wavelength of 3359 cm⁻¹. The Si-O-Si (siloxane) group experienced a slight shift at the 1003 and 529 cm⁻¹ wavelengths. The Al-O-Al group remains at a wavelength of 776 cm⁻¹. The Si-C group was slightly shifted to 691 cm⁻¹. This shift is influenced by the immobilization process of activated carbon on clay. In addition, there is a new peak that appears at a wavelength of 2112 cm⁻¹ which indicates the group of activated carbon, namely C=C alkynes. Then, also appeared alkylhalide groups from activated carbon at a wavelength of 456 cm⁻¹.

Spectrum c shows that the C-H alkyne group at a wavelength of 3212 cm⁻¹, the aromatic C = C group at the wavelengths of 1574, 1557 and 1538 cm⁻¹, and the alkylhalide or haloalkane groups at wavelengths 422 and 417 cm⁻¹. This indicates that activated carbon has been formed from the peel of lal fruit.

4. References
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