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Adsorption of Indigo Carmine Dye and Copper Ion by Kaolin from Bangka Island

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Abstract

Indigo Carmine (IC) is a synthetic dye that might promote irritations, vomiting, and diarrhea when orally consumed in high concentrations. Copper is a dangerous heavy metal that tends to accumulate in organisms' cells as a pollutant. Hyperproduction of IC and accumulation of copper in water could cause an environmental problem that must be adequately addressed. Kaolin is found abundant on Bangka Island. Based on XRD analysis, Kaolin Island from Bangka Island consists of two important minerals, Kaolinite and Rectorite. Kaolin has chemical and physical properties, which make it useful in several applications, including as an adsorbent. This study aims to evaluate how Kaolin can adsorb copper ion from acid wastewater (pH < 0.5) and determine natural Kaolin's ability to adsorb IC dye from an aqueous solution. Many factors, including mixing time and number of doses of Kaolin, influence Kaolin's ability to absorb IC. The longer the stirring time, the more IC is absorbed. The optimal temperature for IC adsorption is 25˚C. The optimal kaolin dose for IC adsorption is 20% (m/v). Copper concentration after application of Kaolin tends to decrease, with optimal at 25.09% (m/v) dosage for 19.23% copper removal.

Keywords: Adsorption, Indigo Carmine, Copper, Kaolin, XRD.

INTRODUCTION

The most abundant clay mineral is kaolinite. Sands or rocks dominantly contain kaolinite are called Kaolin. Kaolin is named after a mountain in Jiangxi Province in south-eastern China called the Gaoling, where this mineral was well-known from old times. Kaolinite's chemical formula is $Al_2Si_2O_5(OH)_4$. Kaolinite's structure is a layered silicate mineral with a tetrahedral silica layer (SiO_4) through oxygen atoms bonded to an octahedral layer of alumina (AlO_6). Kaolin has chemical and physical properties, making it useful in several applications, such as coating and filling paper, plastics, catalysis (E. S. Rahayu et al., 2018), rubber, ink, ceramics, insecticides, etc. and numerous other uses. In Indonesia, Bangka Island is one of the most abundant deposits of Kaolin (Astutiningsih et al, 2018). Kaolin is an ingredient in cosmetics, soaps, polishes, and toothpaste (Shu et al., 2016). In pharmaceutical applications, Kaolin can be given as an excipient, filler, emulsifying, and suspension agent (Sa'adah et al, 2019). Kaolin can be calcined to produce metakaolin. In recent years, metakaolin has been material to produce supplementary cementitious materials (SCM). Metakaolin is an essential ingredient in the most concrete mixture. Another silica-alumina mineral syntheses such as zeolite (Rahayu et al, 2014), amorphous silica alumina (E. S. Rahayu et al., 2018), mesoporous silica (Shu et al., 2016b), or nanocomposite silica (Asriza et al, 2020) can be done by using Kaolin or metakaolin.

Indigo Carmine (IC) is an organic salt dye with the chemical formula ($C_{16}H_8N_2Na_2O_8S_2$). This dye is well dissolved in water. In an aqueous solution IC is blue bright at pH less than 11.4. It is used for wool, hair dyeing, food and beverage coloring, and many more. Some studies show that IC might promote irritations, vomiting, and diarrheal when orally consumed in high concentration by human beings (Lakshmi et al, 2009). High production due to the high demand for Indigo Carmine could cause environmental pollution in water. This problem must be handled properly.

Meanwhile, Kaolin has been known as a dye adsorbent as some reports show Kaolin has a high ability to adsorb dye such as basic yellow dye (Aragaw & Angerasa, 2019) or amido black (Ayanda et al., 2018a). Still, here we aim to evaluate the ability of natural Kaolin collected from Bangka Island to absorb indigo carmine dye. This purpose is the novelty of this research.

Copper is a dangerous pollutant (Irdhawati et al, 2020; Male et al, 2019) that hardly be degraded and tends to accumulate in organisms' cells. In high concentrations, it causes damage to the cells, disrupts metabolisms, and triggers the formation of cancers (Harvey et al, 2016; Irawati & Tahya, 2021). Kaolin was reported as an adsorbent of heavy metal ions like copper, lead, cadmium, chromium, nickel, lead, and zinc (Chantawong et al, 2003). Many reports conduct the adsorption process of copper by Kaolin with a pH above 2. This study examines how Kaolin from Bangka Island can adsorb copper ions from acid wastewater (pH < 0.5).

METHODOLOGY

Materials and Instrumentals

Kaolin was collected from Bangka Island. Indigo carmine (Merck) was used as a synthetic dye for the adsorption assay. The wastewater was collected from one of the laboratories in Tangerang. The Laboratory tools used are analytical balance (Ohaus), furnace (Thermolyne) pH meter (Ohaus) to determine pH. The ICP-OES instrument for determination of copper concentration in solution, XRD, and FTIR for mineral crystal determination and functional groups analysis. UV-Vis DLab SP-V1000 was used to measure the absorbance of IC in solutions. The standard laboratory glasses tools (PYREX and IWAKI) are used throughout the research.

Methods

Preparation of Kaolin

Kaolin was taken from Bangka Island, Indonesia. The kaolin mineral powder was washed with distilled water for twice. After that, it rinsed, and dried in an oven at 85 ^{o}C for 48 hours. The dried Kaolin was ground to powder and sieved. This sample was taken for further characterization with XRD and FTIR.

Indigo Carmine Adsorption Process

Determination of IC standard curve

The IC solutions of 10, 8, 6, 4, and 2 ppm were prepared by diluting 100 ppm IC standard solution using a volumetric flask with distilled water. Take 3 mL of every solution and put it in a cuvette to test the absorbance of the solution at a wavelength of 610 nm (measurement twice).

Effect of stirring time on natural kaolin adsorption ability of indigo carmine

Prepared 500 mL of 10 ppm Indigo carmine (IC) solution by diluting 100 ppm standard solution using a volumetric flask. Transferred 200 mL of 10 ppm IC solution into a 500 mL Erlenmeyer. Take 3 mL of 10 ppm IC solution into a UV-Vis spectrophotometer cuvette and measure the absorbance at a wavelength of 610 nm (2x measure). Add 10.0 g of dry kaolin powder into an Erlenmeyer containing 10 ppm IC solution. Measure the temperature of the mixture with a thermometer. Stir the mixture using a magnetic stirrer and then turn on the stopwatch for 40 minutes from the start of stirring. After every 10 minutes of stirring, take 10 mL of the mixed solution and put it in a centrifuge tube to separate solids and liquids in a centrifuge. After 3 minutes, centrifuge at 5000 rpm, take out the centrifuge tube carefully and transfer it to a tube rack. Take 3 mL of the supernatant (liquid) in a centrifuge tube and put it in a cuvette to test the absorbance of the solution at a wavelength of 610 nm (3x measurements).

Effect of kaolin dosage on indigo carmine adsorption

Prepared 500 mL of 10 ppm IC solution by diluting 100 ppm standard solution using a volumetric flask. Prepare 6 pieces of 250 mL Erlenmeyer flask containers. Label 1 – 6 on the Erlenmeyer flask. Into each Erlenmeyer, pour 80 mL of 10 ppm IC solution. Then add 2 g of Kaolin into Erlenmeyer 2. Add a total of 4 g of Kaolin is added to Erlenmeyer 3. A total of 8 g of Kaolin is put in Erlenmeyer 4. 16 g of Kaolin is put in Erlenmeyer 5, and as much as 20 g of Kaolin is put in Erlenmeyer 6. Erlenmeyer 1 has no Kaolin that added but only 80 mL IC solution. Insert the magnetic bar and cover it with plastic and rubber bands. Stir for 40 minutes with a magnetic stirrer, and record the temperature of each Erlenmeyer after stirring is completed. Take 10 mL of the mixed solution and put it in a centrifuge tube to separate solids and liquids in a centrifuge. After 3 minutes, centrifuge at 5000 rpm. Take out the centrifuge tube carefully, don't shake it! Transfer to a tube rack. Take 3 mL of the supernatant (liquid) in a centrifuge tube and put it in a cuvette to test the absorbance of the solution at a wavelength of 610 nm (3x measurements).

Effect of temperature on indigo carmine adsorption by natural Kaolin

Three pieces of 250 mL Erlenmeyer flasks were prepared and labelled for 1 – 3 on the flask. Pour into each Erlenmeyer 100 mL of 10 ppm IC solution. Add 10.0 g of Kaolin into every flask (label 1-3). Placed it on the magnetic stirrer. Place the closed Erlenmeyer in the designated place for each specified temperature variation. Temperature variations are 6 ^{o}C (in an iced water bath), 25 ^{o}C at room temperature, and 60 ^{o}C (in a water bath). The thermometer was set in every flask to observe the temperature. Stir for 40 minutes with a magnetic stirrer, and record the temperature of each Erlenmeyer after stirring is completed. Take 10 mL of the mixed solution and put it in a centrifuge tube to separate solids and liquids in a centrifuge. After 3 minutes, centrifuge at 5000 rpm, carefully removing the centrifuge tube, do not shake it! Transferred to a tube rack. Take 3 mL of the supernatant (liquid) in a centrifuge tube and put it in a cuvette to test the absorbance of the solution at a wavelength of 610 nm (measurement three times).

Copper ion adsorption by natural Kaolin

The wastewater obtained from the laboratory was 1 L, then diluted by adding 4 L of distilled water so that the total volume of the solution is 5000 mL. The pH of this solution was measured by a pH meter. About 100 mL of this solution was taken for ICP-OES analysis to observe the content of copper. Weight the natural Kaolin for about 1, 5, 20 g and 50 g, and put it into a 500 mL Erlenmeyer flask. Into every flask, add 200 mL of wastewater. Then the stirring was carried out for 5 hours at room temperature, around 27˚C. Filter the mixtures and take 100 mL of each filtrate for the ICP-OES analysis of copper (Irawati & Tahya, 2020). The adsorption percentage was counted with Equation 1.

$$\text{Adsorption (\%)} = (C_o - C_a)/C_o \times 100\% \qquad (1)$$

C_a is the concentration of copper in solution after adsorption (mg/L), C_o is the concentration of copper in solution before adsorption (mg/L).

RESULTS AND DISCUSSION

XRD analysis of natural Kaolin

Natural Kaolin has been characterized by X-ray Powder Diffraction (XRD), and the diffractogram is shown in Figure 1. XRD Quantitative analysis shows the presence of two phases/minerals in Natural Kaolin from Bangka Island. Rectorite mineral is a clay containing a dioctahedral mica and dioctahedral smectite in a 1:1 ratio. This mineral has the advantage of easy cations exchange and is hydrophobic (Dietel et al, 2015). Kaolinite is a silicate mineral consisting of one silica tetrahedral sheet linked to an octahedral alumina sheet through an oxygen atom. Natural Kaolin from Bangka has 55% crystallinity. XRD data confirm the Kaolin from Bangka Island has kaolinite as its main component. Rectorite (He et al., 2022; Yu-bin et al, 2014) and kaolinite (Ayanda et al., 2018b) are clay minerals that can be used as adsorbents.

FTIR analysis of natural Kaolin

FTIR has analyzed Kaolin from Bangka Island; the result is shown in Figure 2, and its interpretation is written in Table 2. Based on the FTIR data, the peak at 698 – 795 cm^{-1} and 1031 – 1115 cm^{-1} corresponded to the Si-O vibration (Tahya et al, 2019). The peak at 537 cm^{-1} corresponds to vibration from the Al-O-Si bond, which affirms that Kaolin from Bangka is an alumina-silica mineral. There is some organic content of the natural Kaolin from Bangka as indicated by the weak peaks of asymmetrical and symmetrical elongation of CH_2 group (Tahya et al, 2023). This phenomenon is expected since this Kaolin was collected from a natural source and treated with simple cleaning and drying processes before application.

Standard curve of Indigo carmine

The standard curve (Figure 3) was made to determine the Indigo Carmine concentration in solution before and after treatment with natural Kaolin. The curve's correlation factor (R^2) is 0.9997 or 99.97%, which means that the correlation of concentration and absorbance is quite strong and linear.

Result of stirring time effect on natural kaolin adsorption ability of indigo carmine

The effect of stirring time on the ability of Kaolin to adsorb IC is shown in Figure 4. It offers an effect of stirring time on the concentration of IC dye, that the longer the stirring time, the smaller or less Indigo Carmine concentration. Kaolin can absorb Indigo Carmine dyes. The decrease in IC concentration occurred because the longer it was stirred, the more dye fascinated by Kaolin's interlayer structure. The adsorption ability of Kaolin is influenced by many factors, one of which is the stirring time.

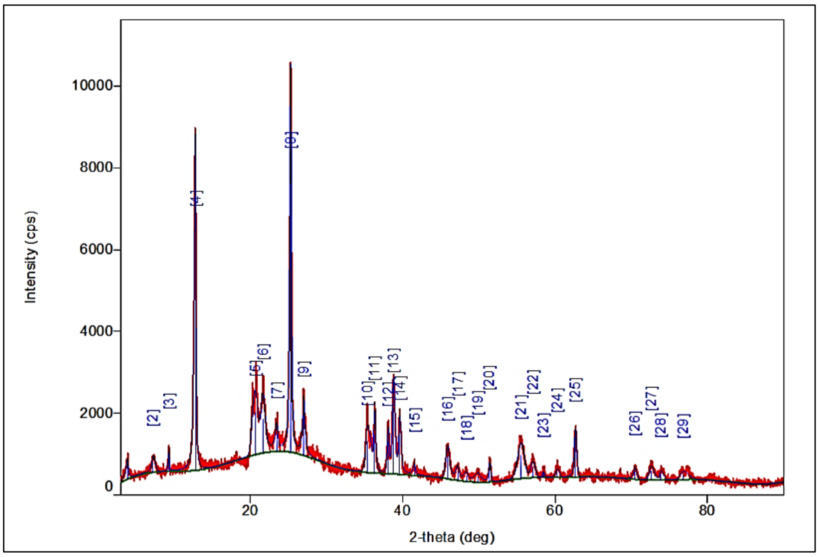

Figure 1. XRD spectrum of natural Kaolin from Bangka

Table 1. The XRD Quantitative analysis result of natural Kaolin from Bangka

Phase name	**Formula**	**Figure of merit**	**Phase reg. detail**	**DB card number**
Rectorite	$NaAl_4(Si,Al)_8O_{20}(OH)_4. 2H_2O$	1.126	ICDD (PDF2.DAT)	00-025-0781
Kaolinite	$Al_{2(}Si_2O_5)(OH)_4$	1.359	ICDD (PDF2.DAT)	00-079-1570

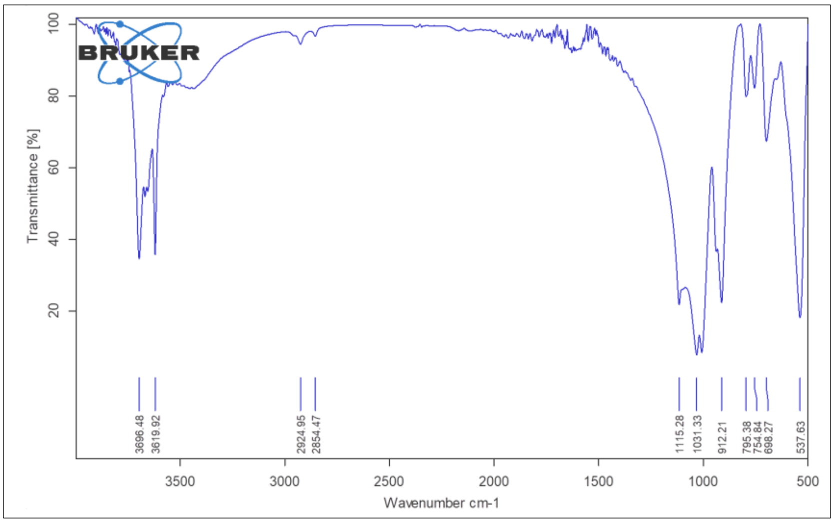

Figure 2. FTIR spectrum of natural Kaolin from Bangka Island

Table 2. Interpretation of FTIR spectrum of natural Kaolin from Bangka Island.

Wavenumber (cm^{-1})	Functional Group
537	Vibration form Al-O-Si bond
698-795	Vibration from Si-O
912	Vibration from Al-OH-Al
1031-1115	Asymmetric stretching vibrations from Si-O bonds
2854	Asymmetrical elongation of CH_2 group
2924	Symmetrical elongation of CH_2 group
3619	Vibration of O-H bonds pointing into the layers towards the tetrahedral sheets (from kaolinite)
3696	Vibration form interlayer O-H groups (from kaolinite)

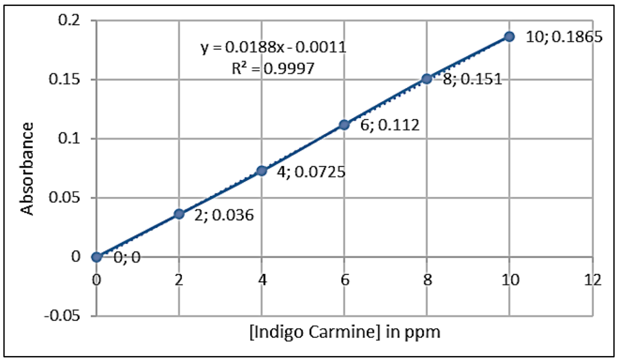

Figure 3. Standard curve for indigo carmine dye.

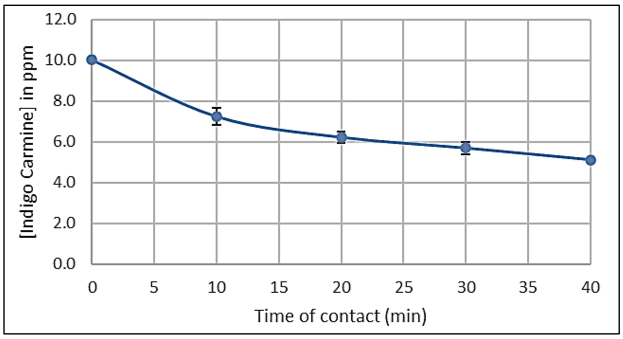

Figure 4. Stirring time effect on natural kaolin adsorption ability of indigo carmine

At a stirring time of 0 minutes, the IC concentration was at its highest value because there was still no kaolin to absorb IC dyes. Stirring causes collisions between the Kaolin and the sample substances. At a stirring time of 10 minutes, a drastic reduction in dye concentration occurred. At 40 min of stirring time, the concentration of IC was the lowest. If the stirring continues, more dye will be absorbed. Finally, the Kaolin reaches a saturation point (no longer able to absorb the dye), and the graph becomes flatter.

Result of kaolin dosage effect on indigo carmine adsorption

Figure 5 shows the effect of the number of doses of Kaolin used in adsorption on the concentration of the Indigo Carmine dye remaining in the solution. The more Kaolin, the more frequent particle collisions occur between the Kaolin and the sample substance in the mixture. The stirring time was 40 minutes, as the previous results obtained for the highest adsorption value on stirring. The ability to absorb Kaolin is influenced by many factors, including the mixing time and the number of doses of Kaolin used. The optimal kaolin dose for IC adsorption is 20% (m/v).

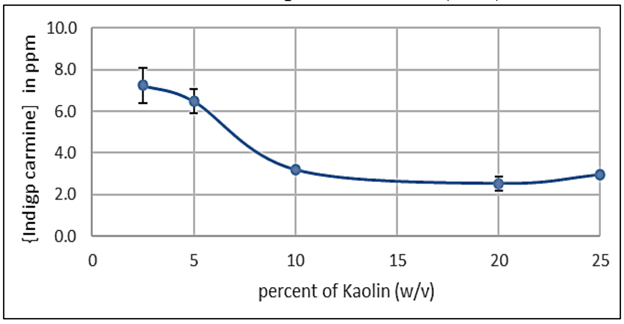

Figure 5. The kaolin dosage influences indigo carmine adsorption.

If too much Kaolin is used, the adsorption ability is not optimal because the kaolin powder in the mixture cannot move properly during the stirring process. At a dose of 25%, the amount of Kaolin is too saturated and reduces the movement of each kaolin particle during stirring, so adsorption does not become optimal.

Result of temperature effect on indigo carmine adsorption by natural Kaolin

The effect of temperature on the adsorption ability of IC by Kaolin is shown in Figure 6. Temperature significantly affects the stirring process and the ability of Kaolin to bind IC in solution. Figure 6 shows the maximum adsorption capability at normal or room temperatures. At a temperature of 25 ˚C, the concentration of IC adsorbed by Kaolin is higher than at other temperatures, and the remaining IC concentration in solution is the least. This data shows that 25 ˚C is the optimum temperature for the Kaolin to adsorb IC, among other observed temperatures.

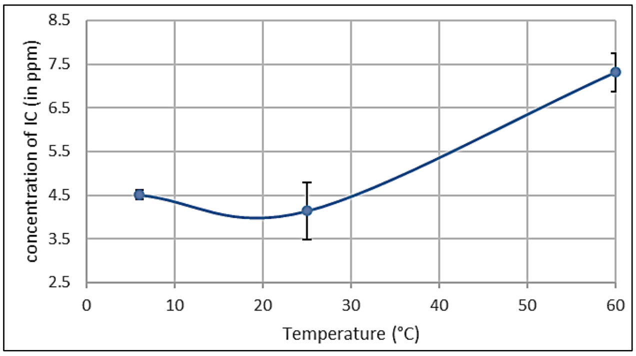

Figure 6. The effects of temperature to the adsorption of IC by Kaolin.

Stirring at a low temperature (6 ˚C) shows the concentration of the solution whose IC remains higher than stirring at normal temperature. While stirring at high temperature (60 ˚C) showed, the concentration of IC solution remaining in solution was much higher than stirring at low and normal temperatures. At 60 ˚C, the IC dye molecules detached from the kaolin interlayer, so the adsorption did not go well.

Copper ion adsorption by natural Kaolin

The ability of natural Kaolin from Bangka to adsorb copper from acidic wastewater was observed, as shown in Table 3. The concentration of copper in the solution after application with Kaolin is decreased. This condition shows that natural Kaolin can bind copper ions in a strongly acidic pH and remove them from the solution. The amount of copper adsorbed in the Bangka kaolin pores increases with increasing doses of Kaolin in the solution.

Table 3. ICP-OES result of copper adsorption by natural Kaolin.

Dosage of natural Kaolin (% m/v)	[Cu] in solution after treatment with natural Kaolin (mg/L)	[Cu] in wastewater (mg/L)	pH of wastewater	Percent of adsorption (%)
0.57	160	182	0.28	12.09
2.57	158	182	0.27	13.19
10.12	167	182	0.28	8.24
25.09	147	182	0.29	19.23

CONCLUSION

The ability of Kaolin to absorb IC is influenced by many factors, including the stirring time and the number of doses of Kaolin used. At 40 min of stirring time, the concentration of IC was the lowest. If the stirring continues, more dye will be absorbed until the Kaolin reaches a saturation point (no longer able to absorb the dye), and the graph becomes flatter. The optimal kaolin dose for IC adsorption is 20% (m/v). 25 ˚C is the optimum temperature for the Kaolin to adsorb IC from the solution. The concentration of copper in the solution after application of Kaolin tends to decrease with optimal at 25.09% (m/v) dosage of Kaolin for 19.23% copper removal. This number shows that natural Kaolin can bind copper ions in a strongly acidic pH and remove them from the solution.

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