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Study on the Effect of Chitosan Concentration on the Synthesis of Epichlorohydrin Crosslinked Charcoal-Chitosan Composite on Its Ability to Adsorb Naphthol Blue Black Dye

Kajian Pengaruh Konsentrasi Kitosan Pada Sintesis Komposit Arang-Kitosan Terikat-Silang Epiklorohidrin Terhadap Kemampuannya Dalam Mengadsorpsi Zat Warna *Naphthol Blue Black*

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ABSTRACT

The synthesis of a charcoal–chitosan composite crosslinked with epichlorohydrin (ACCE) as an adsorbent for naphthol blue black dye was carried out to improve the suboptimal adsorption capacity of activated charcoal. This study aims to characterize the functional groups and water content of the ACCE composite and to investigate the effect of chitosan concentration on its adsorption performance. The ACCE composites were prepared by mixing activated charcoal with chitosan solutions at various ratios, followed by crosslinking with epichlorohydrin. The effects of contact time, pH, and initial dye concentration on the adsorption capacity of naphthol blue black were examined. FTIR analysis of the ACCE composite confirmed the interaction between activated charcoal and chitosan crosslinked by epichlorohydrin, indicated by the appearance of a peak at 2377 cm^{-1}, attributed to N–H bending. The adsorption study showed that equilibrium was achieved at 30 minutes. The pH effect revealed that the highest adsorption percentage was obtained at neutral pH, with the AC8CE2 composite achieving 10.26 mg/g, likely due to enhanced chitosan stability by the crosslinker. Furthermore, increasing the initial dye concentration resulted in higher adsorption capacities, with AC8CE2, AC8CE4, AC8CE6, and AC8CE8 composites reaching 32.86 mg/g, 29.49 mg/g, 26.06 mg/g, and 28.08 mg/g, respectively.

Keywords: Adsorption, activated charcoal, chitosan, epichlorohydrin, naphthol blue black.

ABSTRAK

Sintesis komposit arang-kitosan terikat-silang epiklorohidrin (ACCE) sebagai adsorben zat warna *naphthol blue black* telah dilakukan karena kapasitas adsorpsi dari arang aktif kurang maksimal. Penelitian ini bertujuan untuk mengetahui karakteristik gugus fungsi dan kadar air komposit ACCE, serta mendapatkan data pengaruh konsentrasi kitosan terhadap kemampuan adsorpsinya di dalam komposit ACCE sebagai adsorben zat warna *naphthol blue black.* Komposit ACCE terdiri dari campuran arang aktif dan larutan kitosan dari berbagai variasi rasio yang diikat silang menggunakan

epiklorohidrin kemudian dianalisis pengaruh waktu kontak, pH, dan konsentrasi awal terhadap kemampuan adsorpsi zat warna *naphthol blue black.* Hasil karakterisasi spektra FTIR pada komposit ACCE menunjukkan bahwa telah terjadi interaksi antara arang aktif dan kitosan terikat-silang epiklorohidrin, hal tersebut dibuktikan dengan bilangan gelombang 2377 cm^{-1} yang muncul pada spektrum komposit ACCE yang diidentifikasikan sebagai N-H tekuk. Pengaruh waktu kontak dengan variasi menunjukkan bahwa waktu kesetimbangan tercapai pada waktu 30 menit. Pengaruh pH optimum menunjukkan bahwa persentase adsorpsi paling tinggi terjadi pada pH netral yaitu pada komposit AC8CE2 mencapai 10,26 mg/g, hal tersebut dikarenakan adanya pengikat-silang dapat meningkatkan stabilitas kitosan. Pengaruh konsentrasi awal terhadap kemampuan adsorpsi menunjukkan semakin meningkatnya konsentrasi awal, maka akan meningkatkan kemampuan adsorpsi dari zat warna *naphthol blue black* yaitu pada komposit AC8CE2 mencapai 32,86 mg/g, komposit AC8CE4 mencapai 29,49 mg/g, komposit AC8CE6 mencapai 26,06 mg/g dan komposit AC8CE8 mencapai 28,08 mg/g.

Keywords: Adsorpsi, arang aktif, kitosan, epiklorohidrin, *naphthol blue black*.

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1. INTRODUCTION

The increasing use of dyes in various industries, such as food and textiles, has led to significant environmental problems. Discarded dye waste is particularly concerning as it affects the aesthetic quality of the environment. Moreover, the presence of dyes can inhibit the penetration of sunlight, thereby reducing photosynthetic activity, especially for aquatic organisms living at the bottom of water bodies. Dyes can also become toxic when they interact with metals and chlorine present in the water. This issue is further compounded by the fact that most dyes are resistant to biological degradation, making it necessary to remove or reduce them from aquatic environments. (Fransina, 2008).

Dyes widely used in the textile industry often belong to the azo dye group, which is the largest class of synthetic dyes due to their stability during the dyeing process. One example of an azo dye is naphthol blue black. This dye is commonly used to color fabrics such as wool, nylon, silk, and batik. Naphthol blue black dye is difficult to degrade, especially when its concentration exceeds the permissible limits. According to the Regulation of the Minister of Environment and Forestry of the Republic of Indonesia Number P.16/MENLHK/SETJEN/KUM.1/4/2019, the quality standards for textile wastewater set the maximum allowable BOD at 60 mg/L and COD at 150 mg/L. Moreover, naphthol blue black is non-biodegradable and possesses carcinogenic and mutagenic properties that negatively impact the environment. Therefore, wastewater containing naphthol blue black must be properly treated before being discharged into water bodies (Kusumawardani & Purbasari, 2022).

Several methods have been developed to address environmental pollution caused by liquid organic compounds from textile industry dyes, including coagulation, filtration, and adsorption. Among these, adsorption is recognized as one of the most effective methods for removing color from dye-contaminated wastewater. Various adsorbent materials can be used for this purpose, such as activated carbon and chitosan (Fitriansyah et al., 2021). The first step in achieving an effective adsorption process is selecting an adsorbent with high selectivity and capacity that can also be reused (Fransina, 2008).

Activated charcoal has long been used as an adsorbent for dyes. Research by Hamu et al. (2019) on the use of activated charcoal derived from hazelnut shells (Aleurites moluccana L.

Willd) for naphthol blue black dye removal reported an adsorption capacity of 3.58 mg/g. Activated charcoal possesses a good pore structure and high surface area; however, it lacks abundant functional groups, resulting in predominantly physical adsorption. To enhance its adsorption performance, chitosan can be added to activated charcoal, as chitosan is rich in functional groups such as amine and hydroxyl groups that promote chemical adsorption. Nevertheless, chitosan is unstable under acidic conditions, making it necessary to investigate the synthesis of charcoal–chitosan composites in this study.

Chitosan is a natural polymer with abundant availability; however, it has the drawback of being unstable in acidic conditions, making crosslinking necessary. Chitosan can be crosslinked using agents such as glutaraldehyde (GLA), epichlorohydrin (ECH), ethylene glycol diglycidyl ether (EDGE), and tripolyphosphate (TPP)(Ariyani et al., 2018). The use of epichlorohydrin (ECH) as a crosslinker offers the advantage of targeting the hydroxyl groups of chitosan while preserving its amine groups, which are essential for effective dye adsorption. This crosslinking process is expected to enhance the stability of chitosan in acidic conditions and improve its adsorption capacity (Mardila et al., 2014). Additionally, crosslinking with ECH increases the chemical stability of chitosan, making it insoluble in concentrated acids (pH 1) and bases (Madjid et al., 2018). Based on the above description, this study investigates the effect of chitosan concentration on the synthesis of an epichlorohydrin-crosslinked charcoal–chitosan composite as an adsorbent for naphthol blue black dye.

2. MATERIALS AND METHODS

2.1. Materials

The tools used in this research included an analytical balance (Ohaus), oven (Thermologic), and various glassware (Pyrex), such as beakers, volumetric flasks, Erlenmeyer flasks, volumetric pipettes, dropper pipettes, iron spoons, glass spoons, test tubes, glass funnels, and reagent bottles. Other equipment included a magnetic stirrer, pH meter (DrGray), spray bottle, and analytical instruments such as a Fourier Transform Infrared (FTIR) Spectrophotometer (Platinum ATR Bruker) and a UV-Vis Spectrophotometer (Genesys 10V).

The materials used in this study included chitosan powder derived from shrimp shells (GMP) (moisture content ≤12.0%, ash content ≤1.0%, protein content ≤1.0%, DAC ≥70%), activated charcoal (p.a. Merck, particle size <100 mesh), acetic acid (100%) (E. Merck), sodium hydroxide (NaOH) crystals, concentrated hydrochloric acid (HCl), naphthol blue black dye, epichlorohydrin (analytical grade, Fluka), pH paper, aluminum foil, distilled water, and Whatman No. 42 filter paper.

2.2. Preparation of epichlorohydrin crosslinked charcoal-chitosan composite

In this study, charcoal–chitosan composites were prepared with chitosan solution concentrations of 1%, 2%, 3%, and 4%. To prepare a 1% chitosan solution, 2 g of chitosan powder was weighed using an Ohaus analytical balance, dissolved in 200 mL of 2% acetic acid solution, and stirred until homogeneous. Subsequently, 8 g of activated charcoal was added to the solution and stirred thoroughly. A mixture of 0.5 M NaOH and 1% epichlorohydrin (200 mL) was then added slowly while stirring vigorously. The resulting chitosan gel was allowed to stand for 24 hours, filtered using Whatman No. 42 filter paper, rinsed with distilled water, and dried in an oven at 60°C until a constant weight was achieved. The same procedure was repeated to prepare charcoal–chitosan composites with chitosan concentrations of 2%, 3%, and 4%, each using 200 mL of acetic acid, 8 g of activated charcoal, and 200 mL of the NaOH–epichlorohydrin mixture. The resulting epichlorohydrin-crosslinked charcoal–chitosan composites were labeled as AC8CE2, AC8CE4, AC8CE6, and AC8CE8, corresponding to 1%, 2%, 3%, and 4% chitosan solutions, respectively. Each composite was then analyzed for functional group characteristics using an FTIR spectrophotometer and for moisture content using the gravimetric method.

2.3. Moisture content test

The weight of the empty container and filter paper was recorded prior to use. A total of 0.5 g of the AC8CE2 composite was weighed using an analytical balance and placed into an Erlenmeyer flask. The composite was dried at 105 °C until a constant weight was achieved, then cooled in a desiccator and weighed again to determine the change in mass. The same procedure was carried out for the AC8CE4, AC8CE6, and AC8CE8 composites.

2.4. Preparation of naphthol blue black standard curve

Naphthol blue black solutions with concentrations of 3, 4, 5, 6, and 7 ppm were prepared by diluting a 50-ppm stock solution of naphthol blue black. The absorbance of each solution was then measured using a UV-Vis spectrophotometer at the maximum wavelength of 618 nm.

2.5. Effect of contact time on the ability of epichlorohydrin crosslinked charcoal-chitosan composite to adsorb naphthol blue black dye

A 50 ppm naphthol blue black solution was prepared by diluting a 1000 ppm standard solution using a 5 mL volumetric pipette and transferring it into a 100 mL volumetric flask, followed by dilution to the mark with distilled water. A total of 0.5 g of the dry AC8CE2 composite was weighed using an analytical balance and placed in an Erlenmeyer flask. Then, 100 mL of the 50 ppm naphthol blue black solution was added to the flask. The mixture was shaken using a shaker at 120 rpm for 5, 15, 30, 60, and 120 minutes. After shaking, the composite and filtrate were separated using Whatman No. 42 filter paper, and the filtrate's absorbance was measured with a UV-Vis spectrophotometer. The same procedure was repeated for the AC8CE4, AC8CE6, and AC8CE8 composites.

2.6. Effect of pH on the ability of epichlorohydrin crosslinked charcoal-chitosan composite to adsorb naphthol blue black dye

Acidic, neutral, and basic variations of the 50 ppm naphthol blue black solution were prepared by adjusting the pH using 2% HCl solution to lower the pH and 0.5 M NaOH solution to increase it. A total of 0.5 g of the dry AC8CE2 composite was weighed using an analytical balance and placed in an Erlenmeyer flask, followed by the addition of 100 mL of the 50 ppm naphthol blue black solution. The mixture was shaken using a shaker at 120 rpm for the optimum contact time. The composite and filtrate were then separated using Whatman No. 42 filter paper, and the filtrate's absorbance was measured using a UV-Vis spectrophotometer. The same procedure was repeated for the AC8CE4, AC8CE6, and AC8CE8 composites.

2.7. Effect of initial concentration of epichlorohydrin crosslinked charcoal-chitosan on naphthol blue black

Naphthol blue black solutions with concentrations of 10, 20, 30, and 40 ppm were prepared by diluting a 1000 ppm standard solution and adjusting the pH to the optimum value. A total of 0.1 g of the AC8CE2 composite was weighed using an analytical balance and placed into an Erlenmeyer flask, followed by the addition of 100 mL of the prepared naphthol blue black solution. The mixture was shaken using a shaker at 120 rpm for the optimum contact time. The composite and filtrate were then separated using Whatman No. 42 filter paper, and the absorbance of the filtrate was measured using a UV-Vis spectrophotometer. The same procedure was repeated for the AC8CE4, AC8CE6, and AC8CE8 composites.

2.8. Data Analysis

The water content of the composites was analysed using the gravimetric method. This involved weighing the empty filter paper and then weighing the filter paper containing the ACCE composites after the preparation process. The percentage of water content was calculated using the following equation:

$$Moisture\ content\ (\%) = \frac{initial\ weight\ (g) - final\ weight\ \ (g)}{Initial\ weight\ (g)} \times 100\%$$

The dye adsorption process by the ACCE composites was analyzed using a UV-Vis spectrophotometer at a wavelength of 618 nm.

The absorbance values obtained were converted into concentration values using Microsoft Excel. The percentage (%) of dye adsorbed by the ACCE composite was calculated using the following equation:

$$C_a = C_o - C_e$$

Where,

Ca : Concentration of absorbed substance (ppm)

Co : Initial concentration of solution (ppm)

Ce : Residual dye concentration (ppm)

$$(\%)\ \text{Adsorption} = \frac{Ca}{Co} \text{x } 100\%$$

The data obtained from parameters such as contact time and pH were presented as graphs showing the relationship between the percentage of dye adsorption and contact time, as well as the relationship between the percentage of dye adsorption and pH.

Determination of the amount of adsorbate adsorbed:

The amount of adsorbate adsorbed per unit mass of adsorbent (qeqe, mg/g) was calculated using the following equation:

$$q_e = \frac{Ca\left(\frac{v}{1000}\right)}{m}$$

where,

qe : amount of adsorbed substance (mg/g)

v : volume (mL)

m : mass of adsorbent (g)

3. RESULTS AND DISCUSSION

The initial stage of this study involved the synthesis of a charcoal–chitosan composite crosslinked with epichlorohydrin (ACCE). The purpose of creating this composite was to combine the advantages of both materials—charcoal and chitosan—to enhance dye adsorption efficiency. The composite was prepared by mixing activated charcoal with chitosan, followed by crosslinking with epichlorohydrin. A 2% acetic acid solution was used to dissolve the chitosan, as chitosan is a cationic polymer that is insoluble in water but easily soluble in organic acids. Acetic acid facilitates the dissolution of chitosan into a homogeneous solution (Purwaningsih et al., 2020).

Activated charcoal in powder form was mixed with a chitosan solution and stirred until homogeneous, followed by the slow addition of a mixture of 0.5 M NaOH solution and 1% epichlorohydrin to form an epichlorohydrin-crosslinked charcoal–chitosan gel. This process produces a composite with high adsorption capacity, making it suitable for applications such as dye removal from water or heavy metal adsorption. The formation of crosslinks in chitosan improves its properties, including reduced solubility in water and organic solvents, as well as enhanced mechanical strength of the resulting particles (Sulistyawati et al., 2020). The formation of the epichlorohydrin-crosslinked charcoal–chitosan composite occurs due to interactions between the hydroxyl and carboxyl groups on activated charcoal and the hydroxyl and amine groups on chitosan, as illustrated in Figure 1.

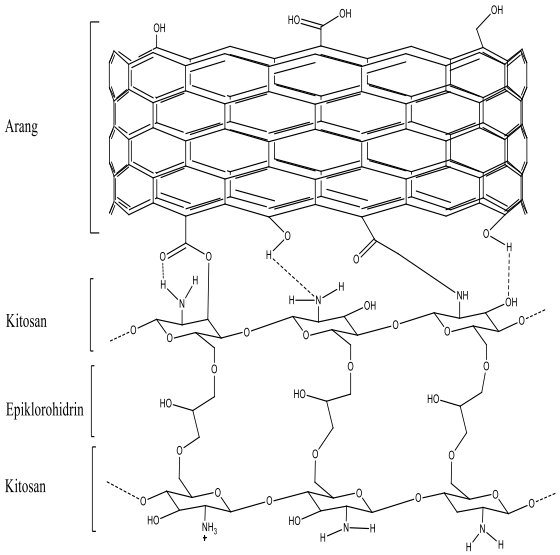

Figure 1. Interaction of activated charcoal, chitosan, and epichlorohydrin

Chitosan dissolves readily in solutions with pH values below 6 and is stable around pH 7, which means it can easily be released or degraded in conditions of pH ≤ 6 when used as an adsorbent. The addition of crosslinking agents can enhance the stability of chitosan under acidic conditions, making it more effective for adsorption applications (Utami *et al*., 2015). Epichlorohydrin crosslinking of chitosan in an alkaline medium occurs through

the formation of covalent bonds between the carbon atoms of epichlorohydrin and the hydroxyl groups of chitosan. This process involves the breaking of the epoxide ring of epichlorohydrin and the release of chlorine atoms, as the reactive sites of epichlorohydrin are located within its epoxide and chlorine groups (Nisfayati et al., 2017). According to Gunawan et al. (2017), crosslinked charcoal–chitosan composites exhibit greater stability compared to non-crosslinked composites. This increased stability is due to the ability of crosslinkers to enhance the mechanical properties of the material, although this may slightly reduce the adsorption capacity.

3.1. Characterization of functional groups of epichlorohydrin crosslinked charcoal-chitosan composites

FTIR spectrophotometric analysis of the composite was carried out to identify the functional group characteristics of the epichlorohydrin-crosslinked charcoal–chitosan (ACCE) composite. The peaks in the FTIR spectrum indicate interactions between charcoal, chitosan, and epichlorohydrin, which are often reflected by shifts in the wave numbers of specific functional groups. The FTIR characterization results of chitosan, activated charcoal, and the ACCE composites are presented in Figure 2.

Based on Figure 6, the infrared spectra display the characteristic peaks of activated charcoal, chitosan, and the ACCE composite, which combines the features of both materials. The functional groups identified from the infrared spectra of activated charcoal, chitosan, and the ACCE composite are summarized in Table 1.

The FTIR spectrum of activated charcoal shows a peak at 2071 cm^{-1}, which corresponds to C=C alkene bonds. The presence of these bonds may result from the activation process of the charcoal and is also observed in the ACCE composite. This suggests that the presence of double bonds in carbon compounds can indicate a high level of purity. Additionally, an absorption band at 1563 cm^{-1} in activated charcoal is identified as an aromatic C=C stretching vibration. This functional group is also present in the ACCE composite, with a slight shift to 1543 cm^{-1}. Such a shift in wave number indicates interactions between the components and suggests that both activated charcoal and the ACCE composite have the potential to adsorb carbon dioxide.

An absorption band at 2377 cm^{-1} was observed in the ACCE composite, which was identified as N–H bending.

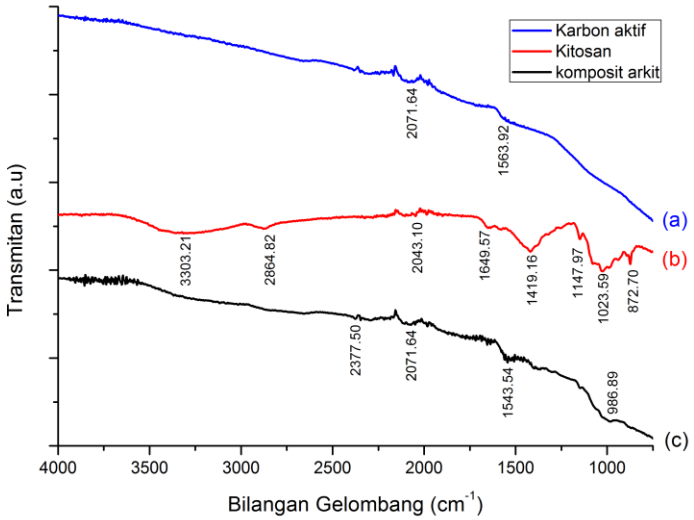

Figure 2. IR spectra of activated charcoal (a), chitosan (b), and ACCE composite (c)

Table 1. Identification of functional groups of activated carbon, chitosan, and chitosan-carbon

Active Charcoal (cm^{-1})	Chitosan (cm^{-1})	ACCE composite (cm^{-1})	Wavelength (cm^{-1})	Functional Group	Reference
	3303		3500-3200	−NH/−OH that is hydrogen bonded	(Sastrohamidjojo, 19
	2864		2850-2950	Unsaturated or Aromatic Alkanes	(Sastrohamidjojo, 20
		2377	2337	N−H bending	(Zaenudin, 2014).
2071	2043	2071	2100-2260	C=C Alkene	(Eso *et al*., 2021).
	1649		1850-1630	Carbonyl (C=O)	(Sastrohamidjojo, 19
1563		1543	1640-1540	C=C aromatic	(Sartika *et al*., 2014).
	1419	1421	1465-1370	C−H bending vibrations	(Andiani *et al*., 2022)
	1147				
	1023		1075-1020	C−O−C Symmetric stretching vibrations	(Sastrohamidjojo, 19
		986	986	C−H bending vibrations	(Silverstein, Bassler,
	872		909-666	Symmetrical stretch N−H	(Sastrohamidjojo, 19

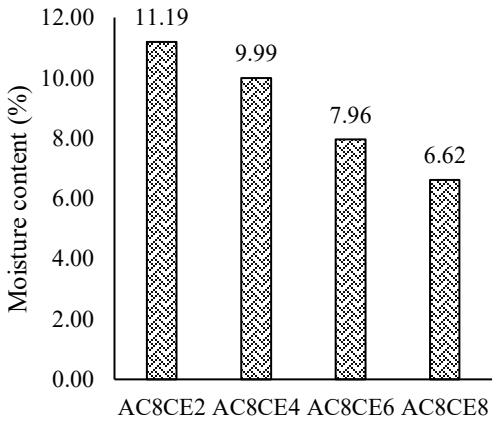

Figure 3. Graph of percentage moisture content at various ratios of epichlorohydrin crosslinked charcoal-chitosan composites

This peak was absent in the spectrum of activated charcoal prior to activation with dyes, indicating the formation of crosslinking between activated charcoal and chitosan.

Additionally, an absorption band at 3303 cm^{-1} appeared in the spectrum of chitosan, attributed to –NH and –OH stretching vibrations involved in hydrogen bonding. This peak suggests the formation of bonds between the hydrocarbon groups in chitosan and epichlorohydrin.

3.2. Moisture Content

The moisture content of the composite was determined using the gravimetric method. This analysis aims to measure the water content by calculating the difference between the initial and final weights of the sample after drying. The gravimetric method involves weighing the sample before and after drying to a constant weight. The effect of chitosan solution concentration on the moisture content of the composite is presented in Figure 3.

Based on the graph in Figure 3, it can be observed that as the concentration of the chitosan solution increases, the moisture content of the composite decreases. This occurs because the pores of the charcoal

become denser with the addition of more chitosan, reducing the amount of water retained within the composite. A lower moisture content enhances the adsorption capacity of the ACCE composite for naphthol blue black dye, as less residual water remains to block the pores (Mudaim & Hidayat, 2021).

percentage of naphthol blue black adsorption by the composites is presented in Figure 4.

Based on the curve in Figure 4, the percentage of naphthol blue black adsorption increases with increasing contact time. The results indicate that the adsorption process reaches equilibrium at an optimum contact time of 30 minutes.

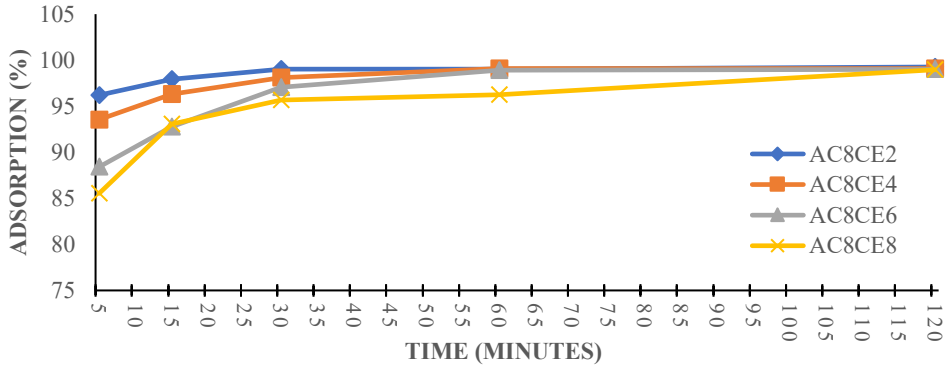

Figure 4. Relationship curve between contact time and adsorption percentage of naphthol blue

The results of this study show that the moisture content of the composites ranges from 6.62% to 11.19%, which complies with the SNI 06-3730-1995 standard that specifies a maximum moisture content of 15% for powder-form adsorbents.

3.3. Effect of contact time on the adsorption ability of naphthol blue black

The determination of adsorption contacts time aims to identify the duration required for the adsorbent and adsorbate to reach adsorption equilibrium. Adsorption equilibrium is achieved when the rate of particle adsorption onto the adsorbent surface equals the rate of desorption (Wulandari *et al*., 2021). Each composite was contacted with a 50 ppm naphthol blue black solution at varying contact times of 5, 15, 30, 60, and 120 minutes, while being shaken at a stirring rate of 120 rpm. The residual concentration of naphthol blue black in the solution after contact with the composite was measured using a UV-Vis spectrophotometer at a wavelength of 618 nm. The adsorption percentage was determined based on the reduction in naphthol blue black concentration before and after adsorption. The relationship between contact time and the

The graph in Figure 4 also shows that higher chitosan concentrations lead to a slower attainment of adsorption equilibrium and a lower overall adsorption capacity. This is likely due to the predominance of physical adsorption, which depends on the pore structure and surface area of the composite. The addition of excess chitosan can block the pores of the activated charcoal, reducing the available surface area for adsorption. At the initial stage of the adsorption process, the pores of the activated charcoal are still open and unoccupied by naphthol blue black dye particles, providing a greater opportunity for the dye molecules to penetrate and be adsorbed. Consequently, the adsorption capacity increases sharply at the beginning until it stabilizes upon reaching the equilibrium condition (Syafrianda *et al*., 2017). After the equilibrium time is reached, the curve becomes linear, indicating that the adsorbed dye is not easily released despite collisions between the composite and water during stirring. According to Sulistyawati (2008), adsorption capacity increases proportionally with time up to a certain point, after which it stabilizes or decreases once equilibrium is achieved.

3.4. Effect of optimum pH

Determination of pH is an important variable in the adsorption process, as it influences the surface charge of both the adsorbent and the adsorbate in the solution. The presence of hydroxyl ions (OH^-) and hydrogen ions (H^+) during the pH adjustment process affects the electrostatic interactions between the composite and the naphthol blue black dye molecules (Hayu *et al*., 2021). In this study, the pH of the naphthol blue black dye solution was varied to represent acidic (pH 3.55), neutral (pH 7.01), and alkaline (pH 9.91) conditions. After contact with the composite, the remaining concentration of naphthol blue black in the solution was measured using a UV-Vis spectrophotometer at a wavelength of 618 nm. The results of this experiment are presented in Figure 5.

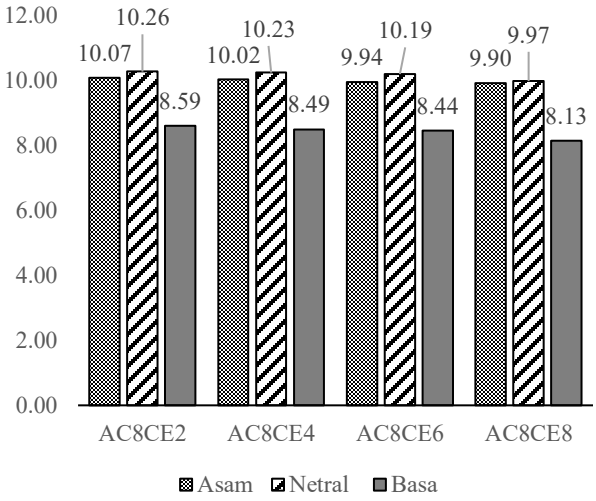

Figure 5. Graph of the effect of pH on the adsorption ability of naphthol blue black on charcoal-chitosan composites.

The graph in Figure 5 indicates that increasing the chitosan concentration in the composite affects its adsorption capacity. The results show that the highest dye adsorption occurs at neutral pH. This is because, under neutral conditions, the amino groups of chitosan can exist in both deprotonated ($-NH_2$) and protonated ($-NH_3^+$) forms, enabling strong electrostatic interactions with the sulfonate groups of the naphthol blue black dye. As a result, the adsorption interaction is maximized at neutral pH compared to acidic or alkaline conditions (Siswati *et al.*, 2014).

The interactions between the dye, activated charcoal, chitosan, and epichlorohydrin are illustrated in Figure 6.

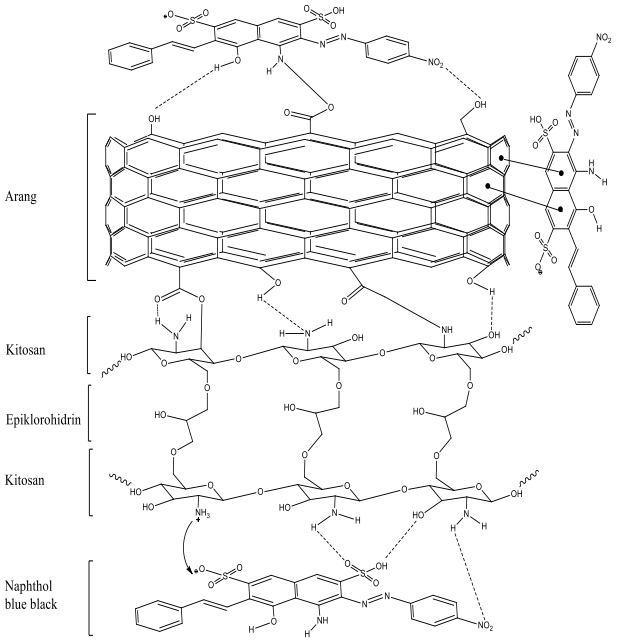

Figure 6. Interaction between ACCE composite and naphthol blue black dye

Under neutral conditions, some of the amine groups ($-NH_2$) in chitosan interact with H^+ ions from the environment, becoming protonated to form $-NH_3^+$ groups. These positively charged groups can then interact with the anionic sulfonate groups of the dye, forming ionic bonds or strong electrostatic interactions that enhance the adsorption process (Pembayun & Rahmayanti, 2020). At acidic pH, the high concentration of H^+ ions in the solution leads to interactions with the hydrophilic groups on the composite surface. This excessive protonation reduces the availability of active adsorption sites, thereby inhibiting the interaction between the composite and naphthol blue black dye. In contrast, at alkaline pH, the naphthol blue black dye tends to interact with Na^+ ions from the base to form salts. Additionally, the presence of OH^- ions can create partial negative charges on the dye molecules,

weakening the electrostatic interactions with the adsorbent (Baunsele & Missa, 2020).

3.5. Determination of adsorption capacity

The determination of the initial dye concentration was conducted to identify the optimum conditions under which the composite exhibits maximum adsorption capacity. Adsorption experiments of naphthol blue black on charcoal–chitosan composites were performed with initial dye concentrations of 10, 20, 30, and 40 ppm at neutral pH and an equilibrium time of 30 minutes.

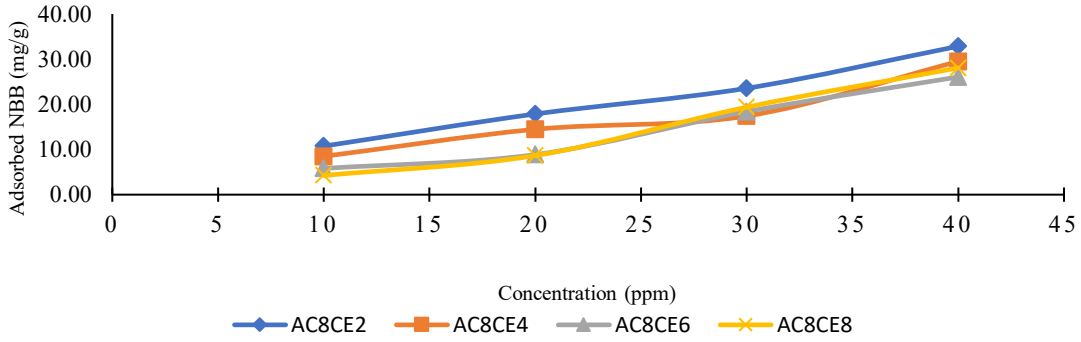

Figure 7. Relationship curve of initial concentration and adsorption ability at equilibrium state (mg/g)

The results, expressed as the amount of naphthol blue black adsorbed per gram of composite (mg/g), are presented in Figure 7, which illustrates the relationship between the initial dye concentration and the adsorption capacity. Based on the graph in Figure 7, the results of this study show that the initial concentration of naphthol blue black dye significantly influences the amount of dye adsorbed by the ACCE composites. As the initial concentration increases, the adsorption capacity also increases. This is because adsorbents possess a finite number of active sites, and each site has an equal probability of binding with adsorbate molecules. If the active sites are not saturated, an increase in the adsorbate concentration results in a greater amount of adsorbate bound to the surface (Utami et al., 2009). In this study, the highest adsorption capacity was achieved at an initial concentration of 40 ppm, with AC8CE2 reaching 32.86 mg/g, AC8CE4 reaching 29.49 mg/g, AC8CE6 reaching 26.06 mg/g, and AC8CE8 reaching 28.08 mg/g. However, at higher concentrations, the adsorption ability of the ACCE composites begins to decrease due to surface saturation, which is likely caused by the increased chitosan content reducing the surface area and pore availability of the composite. Zein *et al.* (2019) in Hayu *et al*. (2021) explains that the adsorption capacity increases with increasing dye concentration due to stronger electrostatic interactions between the active sites on the adsorbent surface and the dye molecules.

4. CONCLUSIONS

The conclusion of this research is:

1. The FTIR spectra of the ACCE composites confirm interactions between activated charcoal and chitosan, as indicated by the appearance of a peak at 2377 cm^{-1}, which is identified as N–H bending. Increasing the concentration of chitosan results in a lower water content within the ACCE composites.
2. The percentage adsorption of naphthol blue black dye by the ACCE composites increased with contact time (5–120 minutes) and reached equilibrium at the optimum time of 30 minutes. A higher chitosan concentration slowed the time required to reach equilibrium and reduced the adsorption capacity.
3. The adsorption of naphthol blue black dye by ACCE composites is influenced by pH, with the optimum adsorption occurring at neutral pH. At this condition, the AC8CE2 composite achieved an adsorption

capacity of 10.26 mg/g. The concentration of chitosan in the composite preparation significantly affects adsorption performance in the presence of crosslinkers.

4. The adsorption capacity of epichlorohydrin-crosslinked charcoal–chitosan composites toward naphthol blue black dye is also affected by the initial solution concentration. At an initial concentration of 40 ppm, the adsorption capacities were 32.86 mg/g for AC8CE2, 29.49 mg/g for AC8CE4, 26.06 mg/g for AC8CE6, and 28.08 mg/g for AC8CE8.

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