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Study Effect of Chitosan-Epichlorohydrin Macropore Beads on Decreasing the Value of Total Dissolved Solid (TDS) and Dyes in Sasirangan Liquid Waste Treatment

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Abstract

Sasirangan liquid waste contains contaminants that are quite dangerous, characterized by high values of total dissolved solids and dyestuffs. This study aims to determine the effect of dose and pH on chitosan-epichlorohydrin macropore beads. The parameters of the measured waste are total dissolved solids and dyestuffs. The process of absorption of sasirangan liquid waste characterizes by FTIR. The results showed that chitosan-epichlorohydrin beads that interacted with sasirangan liquid waste had optimum results at a chitosan dose of 600 mL/L, with a decrease in the total value of dissolved solid by 2161 mg/L and a decrease in dye by 84.78%. Chitosan-epichlorohydrin beads at pH against total dissolved solid gave optimum results at pH 7 with a reduction in the total dissolved solid value of 2269 mg/L, while pH against dyestuffs gave optimum results at pH 3 with a decrease of 94.09%. The interaction between chitosan and chitosan-epichlorohydrin beads occurred a shift in wave numbers from 943 cm^{-1} to 932 cm^{-1} showing the C-O-C group and the chitosan-epichlorohydrin beads spectra with waste at wave numbers 3013-3618 cm^{-1} to 3007-3631 cm^{-1} the O-H vibration group overlapped the N-H vibration and showed the result that the beads Chitosan-epichlorohydrin/adsorb sasirangan liquid waste.

Keywords: Chitosan, beads, crosslinking method, epichlorohydrin, FTIR.

INTRODUCTION

The increase in sasirangan fabric production has a positive impact on MSMEs that produce sasirangan fabric but also has a negative effect, namely the occurrence of environmental pollution due to sasirangan waste being directly disposed of without a processing process. Sasirangan liquid waste is wastewater due to sasirangan industry activities that contain chemical substances or contaminants whose amount exceeds the quality standards of the textile industry liquid waste based on the South Kalimantan Governor Regulation No. 04 of 2007. The sasirangan industry produces liquid waste of an average of 200 liters per day for each (Syauqiah, Nurandini, & Lestari, 2020). Sasirangan liquid waste contains high pollutants and is not qualified to be disposed of in the environment. Many dyes are toxic and endanger living things in the sasirangan industry (Khair, 2017). According to Irawati, Utami, & Hanifa (2011) the value of suspended solids was 862 mg/L. Various methods have been carried out to reduce the quality of dyestuffs in sasirangan liquid waste. There are chemically, physically, and biologically, such as coagulation-flocculation (Rusydi, Suherman, & Sumawijaya, 2017) (Hadiwidodo & Huboyo, 2009) (Haryono, Faizal D, Liamita N, & Rostika, 2018) (Hynes et al., 2020). One of the most frequently used methods of various physicochemical processes is adsorption, adsorption has broad advantages in operational, technical, and convenience (Zhou et al., 2012). Commonly used adsorbent types are activated carbon (A Mizwar & Diena, 2012), and chitosan (Andy Mizwar, 2013) (Sillanpää, Ngah, Teong, & Hanafiah, 2011)

Chitosan is formed into long polymers through β (1,4) bonding of D-glucosamine polysaccharides that have undergone deacetylation from the main component of chitin compounds found in the shell of Crustacea animals such as crabs and shrimp (Salmahaminati, 2022). The adsorption ability of chitosan improves by being modified physically and chemically, such as grafting (addition of active groups), crosslinks (crosslinks), and composites (polymer enhancers). Cross-linking can increase chemical stability, namely insoluble in acids or bases (Madjid, Nitsae, & Sabarudin, 2018). In commonly used chemical modifications, cross-binders are glutaraldehyde, tripolyphosphate, and epichlorohydrin. Epichlorohydrin (EKH) as a cross-binding has the advantage that it does not eliminate the amine group in chitosan. Besides, the cross-link between chitosan and epichlorohydrin occurs covalently, making it more robust and stable (Ariyani, Cahaya, & Mujiyanti, 2018). Based on the description above, it is necessary to conduct research related to synthesizing epichlorohydrin cross-bound macroporous chitosan beads and their effect on the value of dye and TDS levels in sasirangan liquid waste. In addition, the research will study the characteristics of the chitosan-EKH macropore beads functional group before and after interacting with sasirangan liquid waste will be examined.

METHODOLOGY

Materials and Instrumentals

The tools used in this study are standard glassware, analytical balance sheet (Ohaus), spray bottle, pH meter (Yieryi), UV-Vis spectrophotometer (Genesys 10s), TDS meter (HACH SensION^{+} EC5), FTIR spectrophotometer (Shimadzu 8201PC), jerry cans, plastic bottles. The ingredients used in this study were chitosan powder, 99% epichlorohydrin solution, glacial acetic acid (100%), NaOH, $NaHCO_3$, sasirangan liquid waste, aluminum foil, aquacades, and Whatman Filter Paper No. 42.

Manufacture of Epichlorohydrin Cross-Fixing Solution

A total of 80 grams of NaOH is put into a cup glass and dissolved with a small amount of aqueous and stirred until homogeneous, then put into a measuring flask of 1 L and added aqueous to the limit mark.

Effect of dosage on the ability of chitosan-EKH macroporous beads in the treatment of sasirangan liquid waste

Chitosan doses are varied: 300 mL/L, 400 mL/L, 500 mL/L, and 600 mL/L (2% chitosan solution volume per 1 L of sasirangan liquid waste). 2% chitosan solution is taken as much as 150 mL; 200 mL; 250 mL; and 300 mL and added 0.2 grams of $NaHCO_3$, then stirred until foamed. The chitosan solution, still in a foamy state, is put into the burette and then dripped drop by drop into a bowl containing 150 epichlorohydrin solutions (volume of chitosan solution and 1:1 epichlorohydrin solution) and allowed to stand for 24 hours. The formed beads are then filtered and rinsed using aqueous until the pH becomes neutral. After neutral beads, the surface of the beads is dried using dry filter paper. After the surface of the beads is dry, the resulting chitosan-EKH beads are contacted into 500 mL of sasirangan liquid waste by stirring for 5 minutes and allowed to stand for 24 hours. Beads and filtrates are filtered with Whatman paper No.42, and TDS and dye analysis are carried out.

Effect of pH on chitosan-EKH macroporous beads ability to treat sasirangan liquid waste.

The optimum dose of chitosan solution was obtained from the previous procedure used to study the effect of pH. A 2% chitosan solution is added with $NaHCO_3$ by 0.2 grams, then stirred until foaming. The chitosan solution that is still in a foamy state is put into the burette and then dripped drop by drop into a bowl containing the epichlorohydrin solution and allowed to stand for 24 hours. The formed beads are filtered and rinsed using aqueous until the pH becomes neutral. Sasirangan liquid waste is prepared in as much as 1 L. Then the initial pH of the sasirangan liquid waste solution is set to pH 3 to pH 9. The waste solution that has been set to pH is divided into 2. At the optimum dose, on the optimum dose, one part of the liquid waste (500 mL) was contacted with severs. One other part is a negative control without the addition of beads. Both solutions of sasirangan liquid waste are stirred for 5 minutes, allowed to stand for 24 hours, and filtered with Whatman paper No. 42. The resulting filtrate is then measured against TDS and dyestuffs.

Analysis Total Dissolved Solid (TDS)

TDS analysis is carried out based on SNI 06-6989.27-2019 concerning water and wastewater. The cleaned saucer is heated at 180°C for 1 hour in the oven. The cup is then cooled in a desiccator. Samples are taken from 50 mL to 100 mL and fed into a filter equipped with a suction pump and filter paper. After filtering, the filter paper is rinsed as much as 10 mL with aqueous. All filter results, including rinse water, are put into a saucer. The result of the sieve in a saucer is evaporated to dryness. Next, the saucer is put in the oven at 180°C for not less than 1 hour.

Dye Analysis

Analysis of dyestuffs using a Uv-Vis spectrophotometer. Turn on the appliance, and then the maximum wavelength and absorbance are determined. A sample of 2.5 mL was taken into the cuvette. The cuvette is inserted, the bright side facing the light hole of the spectrophotometer.

Analysis Fourier Transform Infrared (FTIR)

The analysis was carried out on chitosan, chitosan-EKH beads, and chitosan-EKH beads samples that had interacted with sasirangan liquid waste. The sample is filtered and oven-dried, then prepared into powder. About 2 mg of the sample was mixed with 200 mg of KBr, homogenized, and then formed into thin pellets by vibrating a mill with a pressure of 8-9 tons. Measurements are made on wave numbers from 400 to 4000 cm^{-1}.

RESULTS AND DISCUSSION

Preliminary Test of Sasirangan Liquid Waste

Preliminary tests on sasirangan liquid waste need to be carried out before interacting with chitosan-EKH macroporous beads to determine the quality of sasirangan liquid waste. The sample is analyzed first as a reference and comparison value. The parameters used are the Total Dissolved Solid (TDS) and dye values. The TDS analysis procedure was carried out based on SNI 06 – 6989.27 – 2019 and for dye values, it was carried out using a Uv-Vis spectrophotometer. The initial pH of sasirangan liquid waste is 7.6. The results of the preliminary test of TDS of sasirangan waste amounted to 2833 mg/L with a quality standard of 2000 mg/L. This shows that sasirangan liquid waste has a fairly high TDS value and exceeds the quality standards set by the government in Kalsel Governor's Regulation No. 04 of 2007. The preliminary test of dyestuffs using a UV-Vis spectrophotometer indicates a high absorbance value of 0.497 at wavelength 521. The liquid waste can be seen in Figure 1.

Manufacture of Chitosan Solution

A 2% chitosan solution was prepared by dissolving as much as 20 grams of chitosan into 1000 mL of 2% acetic acid. The carboxyl group in acetic acid will facilitate the dissolution of chitosan because there is a hydrogen interaction between the amine group derived from chitosan and the carboxyl group in acetic acid. The chitosan-free amine group in the acidic atmosphere will be protonated to form a cationic amino group (NH_3^+) and improve chitosan's polycationic properties (Hambali, Wijaya, & Reski, 2017).

Figure 1. Sasirangan liquid waste

Manufacture of Epichlorohydrin Cross-Fixing Solution

Chitosan is unstable in acidic pH, so in this study, epichlorohydrin was used. The purpose of adding epichlorohydrin as a cross-binding agent is to make the resulting beads durable in acidic and alkaline conditions. The interaction of chitosan and epichlorohydrin is irreversible and can improve chitosan stability. Covalent crosslinking causes strong bonds capable of influencing the mechanical properties of

Effect of dose on the ability of chitosan-EKH macroporous beads to reduce TDS and dye values of sasirangan liquid waste

Dosage is one of the essential parameters for the use of chitosan in the treatment of sasirangan waste. Excessive addition of chitosan doses can cause chitosan to dissolve into wastewater (Benefield, 1982), decreasing the value of TDS and dyestuffs being less effective.

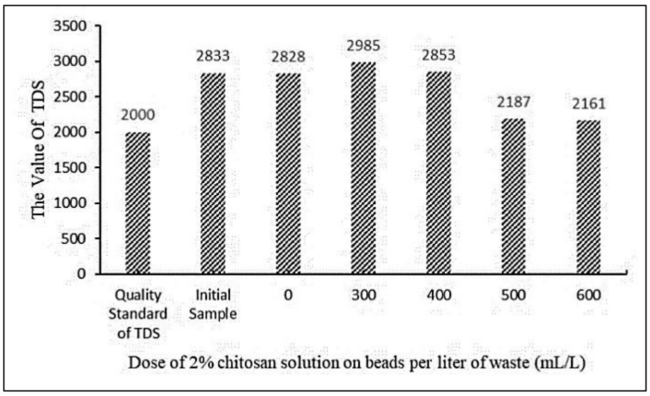

Figure 2. Dose of chitosan-EKH beads on reduced the values of TDS

According to (Laili & Fitri, 2016), Chitosan beads can bind organic and inorganic impurities that are ionized or dissolved in waste. In this study, the highest decrease in TDS value occurred at a chitosan dose of 600 ml/L with a reduction of the TDS value to 2161 mg/L. Although adding chitosan beads can reduce the TDS value of sasirangan liquid waste, the decrease in the TDS value has yet to be maximized because it still exceeds the quality standards set in the South Kalimantan Governor Regulation Number 4 of 2007. The maximum value of TDS is 2000 mg/L. There is an increase in the TDS value at doses of 300 mL and 400 mL higher than the initial sample, which is possible because parts or compounds from the beads are released into the waste at the time of stirring (Figure 2).

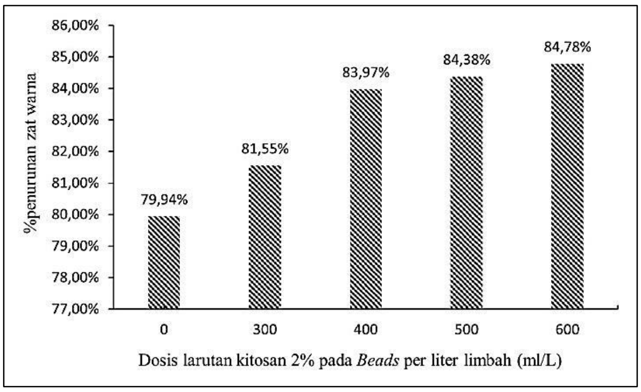

Figure 3. Dose of chitosan-EKH beads on the percentage decrease in dye

In this study, the highest percentage decrease was at a chitosan dose of 600 ml/L, with a percentage decrease reaching 84.78%. The higher the dose added, the higher the percentage decrease in dyestuffs. According to (Chatterjee, Lee, & Wooa, 2010), during the adsorption process, the protonation of the $-NH_2$ group is necessary for interaction with molecules of negatively charged dyestuffs. The absorption mechanism between chitosan beads and dyestuffs involves changing the $-NH_2$ group of chitosan to $-NH_3^+$ in an acidic atmosphere. This group is polycationic, while dyestuffs are acidic when dissolved in water and will be ionized into Na^+ and dye anions. This anion will interact with the NH_3^+ group through electrostatic force (Nugroho, Susatyo, & Prasetya, 2014). According to (Arifin, Karlina, & Khair, 2017), the more chitosan dose, the more significant the decrease in color levels. However, there will be an increase if the solubility of chitosan has been saturated.

Effect of pH on macroporous chitosan-EKH bead's ability to reduce the value of TDS and dyestuffs of liquid waste.

The pH in a system can affect the chitosan functional group, which plays an active role in the process of reducing TDS and dye levels, besides that the pH in the system also affects the shape of substances in sasirangan liquid waste so that in the treatment of liquid waste at an improper pH causes not optimal treatment. Using chitosan-EKH macroporous beads can reduce the percentage of TDS in sasirangan liquid waste samples. With an initial sample value of 2833 mg/L down to 2269 mg/L, there was a 19.91% decrease in efficiency at pH 7 (Figure 3). Under neutral conditions or pH 7, the amine group in chitosan can take the form of a protonation ($-NH_3^+$) and a free amine ($-NH_2$).

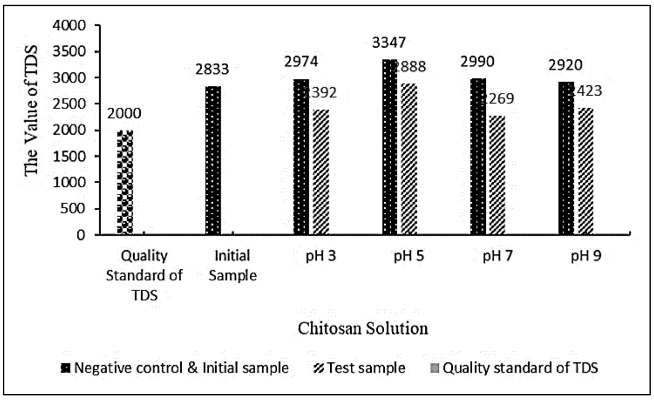

Figure 4. The effect of pH on the decrease in TDS values

This situation allows chitosan beads to bind more TDS-causing impurities to liquid waste samples as negative ions, positive ions, or colloids. While at the acidic pH, the amine group in chitosan will tend to be protonated ($-NH_3^+$) so that the group cannot bind to the substance that causes the TDS, which is positively charged because it will cause the repulsion to resist. Meanwhile, in the alkaline atmosphere, the dominant group in chitosan beads is in the form of a free amine ($-NH_2$) which causes beads to be difficult to interact with dissolved solids in the form of anions.

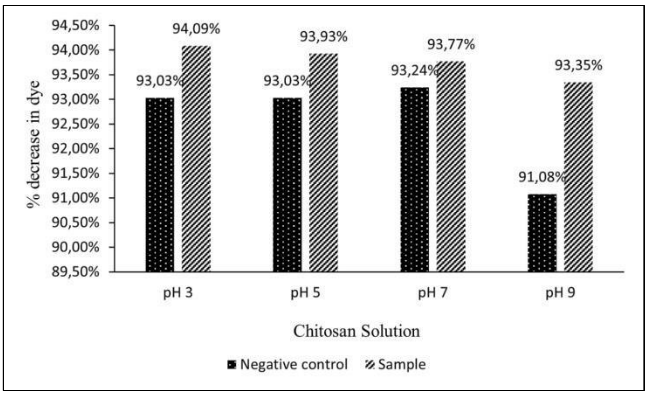

Figure 5. The effect of pH on the decrease in dye

There is an increase in the TDS value at pH 5, which is 2888 mg/L (Figure 4). This occurs because of the part or compound of the beads that are released into the waste at the time of stirring. The negative control in this study was also used as a comparison without adding chitosan-EKH beads. There is an increase in negative control at pH 3 to pH 9. The rise in TDS value in negative control is likely to come from organic and inorganic particles detached from colloidal particles so that they are dissolved in sasirangan liquid waste, which causes an increase in ions in those samples. Adding chitosan-EKH beads is still not yet the maximum because the TDS value still exceeds the predetermined quality standards.

Based on the results of the study, the pH of the optimum solution in reducing parma in sasirangan waste using chitosan-EKH beads was obtained at pH 3 with a percentage reaching 94.09% of its initial concentration before the addition of chitosan-EKH beads. This study's negative controls were comparative and did not interact with chitosan-EKH beads. The pH regulation of the solution affects the adsorbent surface charge, ionization degree, stability, and color intensity of the compounds in the solution (Silva et al., 2015).

According to (Jain, Gupta, Bhatnagar, & Suhas, 2003), under low pH conditions (pH<7), the negative charge on the adsorbent surface can increase protonation and adsorption activity. This allows diffusion to occur so that the active site on the adsorbent surface increases. Thus, the lower the pH of the adsorbate, the more optimal the adsorption process is due to the electrostatic interaction between the negative charge of the dye and the positive charge on the adsorbent surface. In addition to increasing the adsorption ability of H^+ ions in solution, it can also function as a ligand that bridges the bond between adsorbent groups and dye molecules (Maghfiroh, Ulfin, & Juwono, 2016).

Analysis Fourier Transform Infrared (FTIR)

Chitosan analysis using an FTIR spectrophotometer aims to identify the functional group characteristics of chitosan-EKH beads before and after interacting with sasirangan liquid waste (Figure 5). The basic principle of infrared spectroscopy is the interaction between the vibrations of bound atoms/functional groups in molecules through the absorption of electromagnetic radiation of infrared waves. The absorption of infrared radiation can cause a change in dipole moment due to vibration (Mujiyanti, Ariyani, & Lisa, 2021)

The shift in wave numbers occurs from the vibration of the β 1.4-glycosidic in wave numbers 663 cm^{-1} from chitosan to 656 cm^{-1} and 675 cm^{-1} in chitosan-EKH beads and chitosan-EKH beads with waste this identifies the breaking of the β 1.4-glycosidic bond when dissolving chitosan with acetic acid (Dompeipen, 2017) (Rahayu, Tanasale, & Bandjar, 2020). The change in the wave number from the C-O-C group at the wave number 943 cm^{-1} in chitosan to 932 cm^{-1} in chitosan-EKH beads explains the presence of epichlorohydrin binding to chitosan (Nisfayati, Rahmi, & Marlina, 2017), the absence of uptake in chitosan-EKH beads with waste can mean that the molecule or compound does not have such a group. The peak uptake on wave number 1135 cm^{-1} of chitosan to 1168 cm^{-1} in chitosan-EKH beads and chitosan-EKH beads with waste according to the stretching vibration of the C-O group. This indicates that EKH reacts with chitosan hydroxyl carbon atoms and forms covalent bonds, causing the detachment of the EKH epoxide ring and releasing chlorine atoms (Jawad, Abdulhameed, Reghioua, & Yaseen, 2020).

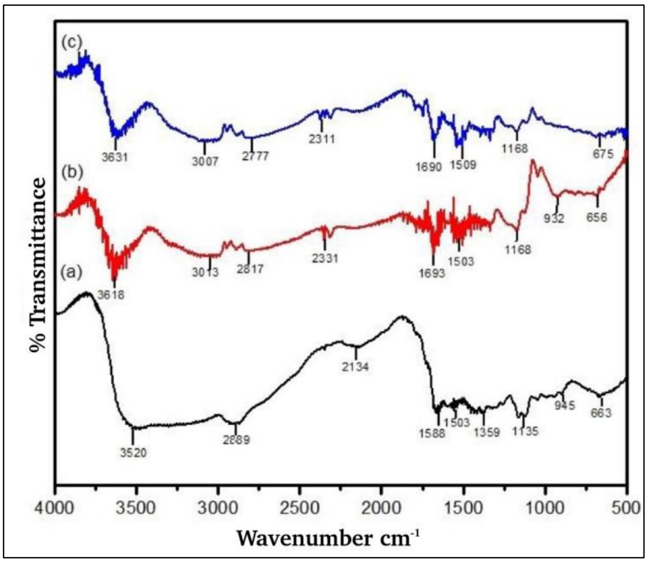

Figure 6. (a) FTIR chitosan spectra, (b) chitosan-EKH beads, (c) chitosan-EKH + waste beads

The peak of chitosan uptake at wave number 1359 cm^{-1} is the stretching vibration of C-N. The wave numbers 1503 and 1588 cm^{-1} in chitosan became 1503 cm^{-1} in chitosan-EKH beads and 1509 cm^{-1} in chitosan-EKH beads, with waste being N-H bending vibrations. It can be indicated that there is no involvement of the amine group in the crosslinking reaction (Jóźwiak, Filipkowska, Szymczyk, Rodziewicz, & Mielcarek, 2017). New absorption peaks appeared in 1693 cm^{-1} and 1690 cm^{-1} in chitosan-EKH beads and chitosan-EKH beads, with waste constituting the C=O stretch vibration of the carbonyl group (Igberase, Ofomaja, & Osifo, 2019). Subsequent uptake at wave numbers 2134-2889 cm^{-1} of chitosan indicates the presence of symmetrical stretching vibrations of aliphatic C-H functional groups, the emergence of new uptakes at wave numbers 2331-2817 cm^{-1} from chitosan-EKH beads and 2311-2777 cm^{-1} from chitosan-EKH beads with waste indicating that a crosslinking process has occurred in chitosan indicates the interaction of chitosan-EKH beads with waste (Gunawan, Shofiyani, & Zaharah, 2017). When viewed in Figure 6 in chitosan, there is a strong and abundant O-H group, so when viewed again on beads, chitosan-EKH and chitosan-EKH beads with their O-H group waste are reduced.

CONCLUSION

The beads chitosan-EKH macropores can reduce the value of TDS and dyestuffs in sasirangan liquid waste. The higher the dose added, the more significant the decrease in TDS and dyestuffs, with a decrease in values of 2161 mg/L in TDS and 84.78% in dyes at an optimum dose of chitosan 600 mL/L. In TDS, with a decrease in TDS values of 2269 mg/L at pH 7, while the decrease in the percentage of dyestuffs reached 94.09% at pH 3. Based on the characterization of FTIR shows the interaction between chitosan, chitosan-EKH beads at wave numbers 943-932 cm^{-1} vibration bending C-O-C and spectra beads chitosan-EKH and spectra beads chitosan-EKH with waste at wave numbers 3013-3618 cm^{-1} to 3007-3631 cm^{-1} O-H vibration groups overlap N-H vibrations, showing that beads chitosan-EKH can adsorb sasirangan liquid waste.

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