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Research Article

Combined absorbent of corn husks and eggshells activated by sodium hydroxide as an adsorbent for Remazol Yellow FG dye in textile waste

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Abstract
Reactive dyes such as Remazol Yellow FG are contaminants that are difficult to degrade in the environment. The adsorption method is an alternative to overcome this problem. Corn husk and eggshells can be adsorbents in adsorption. Corn husk contains an organic compound called cellulose, while eggshells have the main component of calcium carbonate. Corn husks and eggshells have the potential to be adsorbents with different characteristics. This research aimed to determine the optimum conditions for the combined adsorbent of corn husks and eggshells, which included composition comparison, contact time, pH, and application in dye waste. The interactions between Remazol Yellow FG dye in solution and combined adsorbent were conducted in batch method. Based on the research results, the optimum composition of corn husks and eggshells was 1:2, with an optimum contact time of 80 minutes at a pH of 2, resulting in an adsorption capacity of 0.559 mg/g, with an adsorption percentage of 33.95%. Adsorption of Remazol Yellow FG in sewage using combined adsorbents under optimal conditions obtained an adsorption capacity of 36.6579 mg/g with an adsorption percentage of 27.87%. The results demonstrate that the combined adsorbent of corn husks and eggshells is a potentially useful for removing Remazol Yellow FG dye from textile wastewater.

Keywords: Adsorption, Activator, Corn husks, Eggshells, Remazol yellow FG

INTRODUCTION

The development of the textile industry in Indonesia is very rapid. The textile industry produces products in the form of clothing or other clothing materials. Textile dyes are one of the main ingredients used to color fabrics to make the products more attractive. In the process of dyeing fabrics, the excess dye used will be discharged into the environment as waste. Textile industry waste is an environmental problem that must be addressed. According to Lolo & Pambudi (2020), Indonesia produces organic waste of 883 tons/day, and 29% of this waste comes from textile industry waste. The dyeing process in the textile industry is known to leave 10-15% of the dye in the wastewater and will be discharged into the environment as waste (Sukmawati *et al*., 2014). In the textile industry, the synthetic dyes that are often used are reactive dyes of the azo group, one of which is Remazol Yellow FG. This dye is generally used in the batik textile industry and in the dyeing process. Remazol Yellow FG is an azo dye that has bonds (-N=N-) with a complex structure and is stable, so it does not fade easily and is difficult to degrade in the environment (Ariguna *et al*., 2017). One method that can be used to overcome this problem is adsorption (Sausan *et al*., 2021).

Adsorption is a method with the working principle of adsorption of material from a fluid or gas component in the inter-phase area; the material to be separated will later be bound to the surface of the adsorbent (Ifa *et al*., 2021). In the adsorption process, the ability of the adsorbent to adsorb a substance can be increased through the activation process. Activation using an activator such as NaOH can dissolve impurities covering

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the pores so that the ability of the adsorbent to absorb the adsorbate increases (Sihotang, 2021). Adsorption of dyes can be carried out using adsorbents made from organic materials, such as agricultural and livestock wastes, to replace activated carbon, which is considered quite expensive.

Organic waste that can be used is corn husks and eggshells. Corn husk is a residual agricultural product that can be used as an adsorbent because it has a fairly high cellulose content of 36.81% (Sari, 2019). The OH group contained in cellulose and hemicellulose on corn husk can bind to the adsorbate as vinyl sulfone groups from reactive dyes so that the adsorbent can absorb the adsorbate in the form of dyes. Eggshell is a waste that has not been used much and has the potential as an adsorbent. This is because the main constituent of eggshells is calcium carbonate ($CaCO_3$). According to Yuwanta (2010), the $CaCO_3$ content in the eggshell reaches 98.43%. The high calcium carbonate content in the eggshell causes the eggshell to have the potential as an absorbent or adsorbent (Maslahat *et al*., 2015). Eggshell is known to have 7,000 to 17,000 pores, making it an attractive material to employ as an adsorbent (William and Owen, 1995), which can absorb a material such as dye in textile waste.

The use of organic materials as adsorbents can be developed by combining materials. Both corn husks and eggshells have the potential as adsorbents so that they can be combined to increase the adsorption ability of a material. The purpose of this study was to determine the optimum conditions of the adsorbent for the combination of corn husks and eggshells, including composition comparison, contact time, and pH of the solution, as well as to determine the ability of the adsorbent to adsorb waste containing Remazol Yellow FG.

MATERIALS AND METHODS

Materials

The materials used in this study included corn husks obtained from corn traders in the Denpasar area, and eggshells from the Pie Susu factory in Denpasar, NaOH, HCl, dye Remazol Yellow FG, distilled water.

Instrument

Magnetic stirrer, UV-Vis spectrophotometer, FTIR Shimadzu IR prestige-2, oven, analytical balance, pH meter, 80 mesh sieve, filter paper, and a set of glassware

Methods

Production of corn husks and eggshell adsorbents

Corn husks and eggshells are processed separately. The first step was to wash the corn husks and eggshells, then cut the corn husks into small pieces. Corn husks and eggshells were then air-dried. Dry corn husks and eggshells were then ground using a blender to become powder and sieved using an 80 mesh sieve. Corn husks and eggshells powder measuring 80 mesh were then activated using 0.5 M NaOH for 1 hour, then the corn husks and eggshells powder was filtered and rinsed using distilled water until the pH was neutral. Neutral corn husk and eggshell powders were then dried in an oven at 100^{o}C for 2 hours.

Determination of adsorbent optimum conditions

Corn husks and eggshell adsorbents were combined with ratios of 1:0, 0:1, 1:2, 2:1, and 1:1. The combined adsorbent for each ratio was weighed as much as 0.75 g. Then, the combined absorbent for each ratio was used to adsorb 50 mL of Remazol Yellow FG 30 mg/L solution. The adsorption process was carried out using a magnetic stirrer for 60 minutes, then the mixture was filtered and the absorbance of the adsorption filtrate was measured at the maximum wavelength of Remazol Yellow FG, namely 422 nm. The absorbance value obtained was then used to determine the absorbed dye concentration so that each variation's adsorption capacity could be determined. The adsorption capacity was determined by the following formula:

$$q_e = \frac{(C_i - C_f)}{m} \times V \qquad \text{Eq. 1}$$

Where,

q_e = absorbtion capacity

C_i = initial concentration (mg/L)

C_f = final concentration after absorbtion (mg/L)

m = mass of absorbent (g)

V = volume of sample (L)

Determine the optimum contact time

The combined adsorbent with the optimum composition ratio was then used to adsorb 50 mL of Remazol Yellow FG 30 mg/L solution. The adsorption process was carried out using a magnetic stirrer with varying contacting times of 20, 40, 60, 70, 80, 90, and 100 minutes, then the mixture was filtered and the adsorption filtrate was measured for its absorbance at the maximum wavelength of Remazol Yellow FG, namely 422 nm. The absorbance value obtained was then used to determine the absorbed dye concentration so that each variation's adsorption capacity can be determined.

Determine optimum pH

Determination of the optimum pH using combined adsorbents in the optimum composition ratio to adsorb 50 mL of Remazol Yellow FG 30 mg/L solution, which was adjusted for pH conditions with variations of 1,2,3,4,6,8,10,12 then stirred using a magnetic stirrer for optimum contact time. The absorbance value ob-

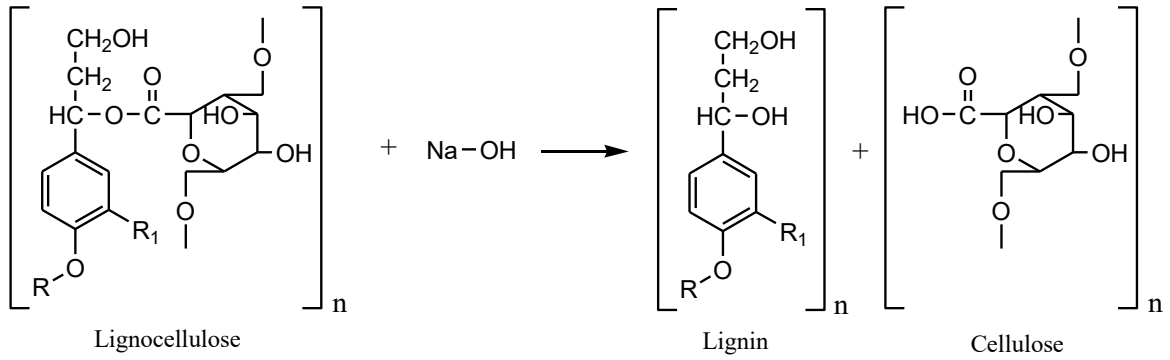

Fig.1. *Bond-breaking reaction lignocellulose by sodium hydroxide (Fegel & Wegeneer, 1995)*

tained was then used to determine the absorbed dye concentration so that each variation's adsorption capacity could be determined.

Applications in textile waste

Textile waste containing Remazol Yellow FG was obtained from a dyeing factory in the Denpasar area. The dye waste was first filtered using filter paper; then, the absorbance was measured using a UV-Vis spectrophotometer to determine the initial concentration of Remazol Yellow FG in the waste. For waste whose initial concentration is known, pH was adjusted by adding NaOH or HCl until it reached the optimum pH condition. Then, the waste was adsorbed using a combined adsorbent in optimum mass ratio and the adsorption process was carried out using a magnetic stirrer during optimum contact time. In the final step, the mixture was filtered and the absorbance of the adsorption filtrate was measured at the maximum wavelength of Remazol Yellow FG, which was 422 nm. The absorbance value obtained was then used to determine the absorbed dye's concentration to determine the adsorbent's adsorption capacity.

RESULTS AND DISCUSSION

Corn husks and eggshell adsorbents

Corn husk of as much as two kg in wet condition and eggshell of as much as one kg were dried and ground, then sieved using an 80 mesh sieve to result in 500 g of corn husk and 800 g of eggshell powders. The resulting adsorbent powders are known to have a moisture content of 7.17% and 4.38% for corn husk and eggshell, respectively. The water content value obtained is to the requirements for the moisture content of a simplicial, which is less than 10% (Ministry of Health, Republic of Indonesia, 2017). The dry corn husks and eggshells' powders were activated using sodium hydroxide 0.5 M as an activator. It can release lignin from lignocellulose, which can interfere with the adsorption process. The bond-breaking reaction lignocellulose by sodium hydroxide served is given in Fig. 1.

Sodium hydroxide as an activator not only functions to release lignin from cellulose but can also dissolve impurities in the adsorbent's pores to increase the adsorption capacity of eggshell powder and corn husk powder. The adsorbent has been activated using NaOH and will go through a rinsing process using distilled water to clean the adsorbent from other compounds as non-cellulose components that can interfere with the adsorption process.

Determination of adsorbent optimum conditions

Optimum composition ratio

The optimum composition ratio determined using 0.75 g of combined adsorbent in various composition ratios of corn husks and eggshells, namely 1:0, 0:1, 2:1, 1:2, and 1:1 are mentioned in Fig. 2, showing that eggshells had a greater adsorption capacity than corn husk. This is seen from the 0:1 ratio, which had a greater adsorption capacity than the 1:0 ratio, which was 0.024 mg/g with a decreasing percentage until 1.17%. The low absorption capacity of corn husk was caused by the nature of Remazol Yellow FG, an anionic dye which has negatively charged, so it can not interact with the OH group in the corn husk, also negatively charged. The 1:1 ratio has the smallest adsorption capacity compared to the other four ratios. These are presumably because corn husks have a lower absorption capacity that can interfere with the absorption process of eggshells. The 1:2 ratio shows a higher adsorption capacity than the other four ratios, which is 0.051 mg/g with a decreasing percentage of 2.47%. These caused the eggshell to have a high $CaCO_3$ content and a lot of pores. $CaCO_3$ is a polar compound that will make it easier to bond with dyes, which are also polar compounds (Lestari *et al*., 2021).

Optimum contact time

The optimum contact time was determined using 0.75 g of combined adsorbent in a 1:2 ratio of corn husks and eggshells to adsorb Remazol Yellow FG in various contact times. The absorption capacity of the combined absorbent with each contact time is mentioned in Fig.

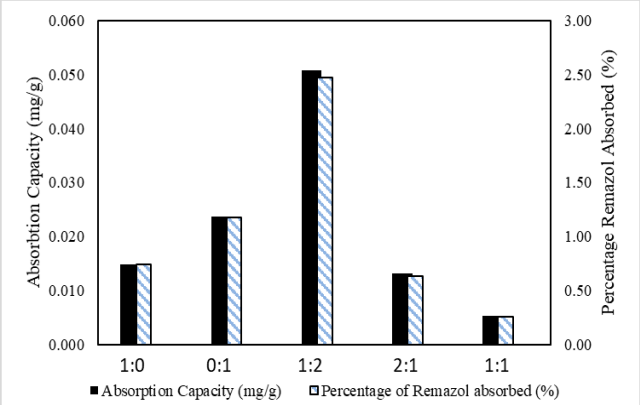

Fig. 2. *Absorption capacity and percentage of Remazol absorbed of the combined absorbent in each composition ratio*

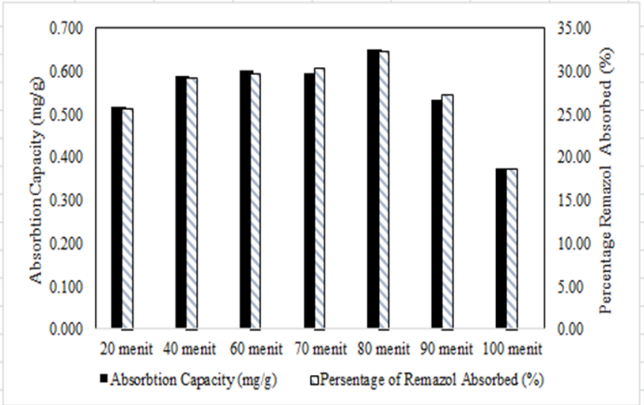

Fig. 3. *Absorption capacity and percentage of Remazol absorbed of combined absorbent with each contact time*

3, showing that with the increase of contact time from 20 to 80 minutes, the adsorption capacity tends to increase, while at 80 to 100 minutes, the adsorption capacity tends to decrease. The data shows that the ability of the combined adsorbent to absorb Remazol Yellow FG increased in line with the increase in contact time until 80 minutes. The adsorption process of Remazol Yellow FG by the combined adsorbent within 100 minutes had the smallest adsorption capacity compared to the other six time variations. These indicated that the combined adsorbent had reached its saturation point, resulting in a desorption process due to reduced active sites on the surface of the adsorbent (Kurniawati and Indriyanti, 2021). The adsorption process at 80 minutes had the highest adsorption capacity compared to other six-time variations, which was 32.33% for the colorant reduction with an adsorption capacity of 0.650 mg/g.

Optimum pH

Optimum pH conditions were determined by the adsorption of Remazol Yellow FG solution, using a combined absorbent of corn husks and eggshells in a ratio of 1:2, with a contact time of 80 minutes in various pH conditions. The research results related to the optimum pH conditions are shown in Fig. 4, showing that the adsorption capacity of the combined adsorbent had decreased significantly when the pH of the solution was above 6, so it shows that the adsorption process of Remazol Yellow FG using a combination of corn husk and eggshell adsorbents gives good results at low pH conditions (acidic). This condition is due to the protonation process in the adsorbent causing electrostatic interactions between the adsorbent and the anionic dyes (Sukarta, 2020). Remazol Yellow FG is an anionic dye that releases Na^+ ions in water to form a negatively charged SO_3^- group (Imaniah *et al*., 2017).

The adsorption process of Remazol Yellow FG by combined adsorbent of corn husks and eggshells at low pH conditions begins with the protonation process of the OH^- group contained in the adsorbent to become H_2O^+. The protonation process will cause the surface of the adsorbent to become positively charged so that later it can bind to the negatively charged sulfonate groups of the dye (Kurniawati & Indriyanti, 2021). In addition, the $CaCO_3$ compound in the eggshell will also undergo hydrolysis to form Ca^{2+} and CO_3^{2-}. Panduragan *et al.* (2018) stated that in low pH (acidic), the eggshell adsorbent tends to become positively charged. It causes a strong electrostatic attraction between the absorbent surface and the negative charge of the dye.

Application in textile waste

The textile waste coming from the dyeing process residue containing Remazol Yellow FG from a textile factory in the Denpasar area showed that the concentration of this dye was 1972.835 mg/g with a pH of 9.7. Under optimal conditions, the combined adsorbent of corn husks and eggshells adsorbed 50 mL of textile waste containing Remazol Yellow FG with a contact time of 80 minutes. The adsorption process was carried out in optimum pH (pH 2) and the initial waste pH (pH 9.7) as a comparison (Table 1). This showed that the adsorption process of Remazol Yellow FG in textile waste using combined adsorbents gave better results at a pH of 2. The combined adsorbent of corn husks and eggshells adsorbed as much as 549.869 mg/L of Remazol Yellow FG from wastewater with an adsorption capacity

Table 1. Absorption capacity of combined absorbent to Remazol Yellow FG in textile waste of natural and optimum pH.

pH	Adsorbed Concentration (mg/L)	Remazol Adsorbed (%)	Absorbtion Capacity (mg/g)
9.7	78.740 ± 11.811	3.99	5.249
2.0	549.869 ± 9.900	27.87	36.658

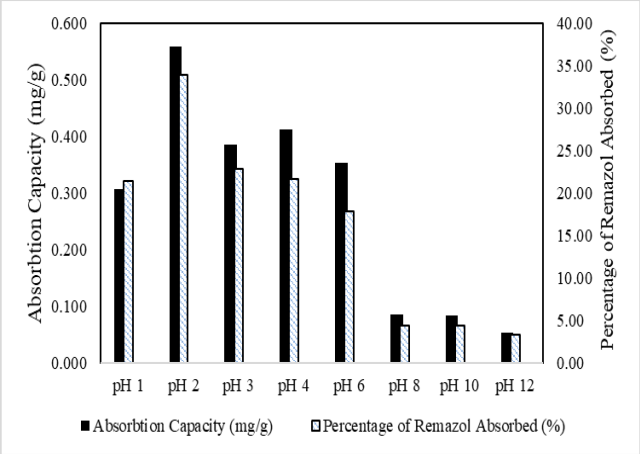

Fig. 4. *Absorption capacity and percentage of Remazol absorbed of combined absorbent in various pH*

of 36.658 mg/g. It shows that the combined adsorbent of corn husks and eggshells under optimum conditions can increase the adsorption capacity to 31.409 mg/g and absorbed dye to 471.129 mg/L. The adsorption of Remazol Yellow FG in wastewater has been carried out by several researchers, such as Herawati *et al*. (2020) using banana blossom as an adsorbent for Remazol Yellow FG in batik wastewater at a pH of 9, a decrease in the concentration of the dye substance was obtained by 4.93%, and Rahmawati *et al*. (2003) using water hyacinth activated by NaOH as an adsorbent for Remazol Yellow FG in textile wastewater at a pH of 2 was obtained an adsorption percentage of 26.506% and an adsorption capacity of 4.44 mg/g. This showed that the combined adsorbent of corn husks and eggshells is in optimum condition and has a better adsorption capacity to Remazol Yellow FG dye from textile wastewater than the adsorbents of banana flower and water hyacinth.

Conclusion

The present study concluded that optimum conditions for the combined adsorbent of corn husk and eggshell obtained at a 1:2 ratio, a pH of 2, and a contact time of 80 minutes with a percentage of the adsorbed Remazol Yellow FG dye of 33.95% and an adsorption capacity of 0.559 mg/g. Using the combined adsorbent of corn husks and eggshells in a ratio of 1:2, under optimum conditions in the textile waste, was able to adsorb 27.87% of the dye with an adsorption capacity of 36.658 mg/g. The combined adsorbent of corn husks and eggshells can be a potentially useful material for removing Remazol Yellow FG dye from textile wastewater.

Conflict of interest

The authors declare that they have no conflict of interest.

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