# RESEARCH ARTICLE



# Performance of Ceramic Membrane Modified with Corncob Activated Carbon for Efficient Remazol Red Removal in Batik Wastewater

(Performa Membran Keramik yang Dimodifikasi dengan Karbon Aktif Tongkol Jagung untuk Penghilangan Remazol Red yang Efisien pada Limbah Cair Batik)

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# **ABSTRACT**

A ceramic membrane modified with corncob activated carbon (CMCC) has been successfully prepared and used to reduce the concentration of remazol red in batik wastewater. This research aims to study the effect of corncob activated carbon (CC) in the ceramic membrane on the porosity of the ceramic membrane and its ability to reduce the concentration of remazol red dye in batik wastewater. The variation of % CC to the mass of clay used was 0%, 5%, 10%, 15%, 20%, and 25%. Characterization of CC includes ash content and characterization of CMCC includes porosity test, morphological analysis, and elemental distribution based on SEM-EDX. The results showed that the ash content of CC before and after activation was 5.625% and 2.974%, respectively. The CMCC porosity test results showed that the more CC added, the greater the porosity of the ceramic membrane. The elemental composition in CMCC is dominated by O, Si, Al and C. The addition of 10% CC obtained the optimum composition on a ceramic membrane. % removal of remazol red is 83.9%. The COD values of batik wastewater before and after processing with CMCC were 484.286 and 26,339 mg/L, respectively.

Membran keramik yang dimodifikasi dengan karbon aktif tongkol jagung (CMCC) telah berhasil dibuat dan digunakan untuk mengurangi konsentrasi remazol red dalam air limbah batik. Penelitian ini bertujuan untuk mempelajari pengaruh penambahan karbon aktif tongkol jagung (CC) pada membran keramik terhadap porositas membran keramik dan kemampuannya dalam menurunkan konsentrasi zat warna remazol red pada limbah cair batik. Variasi % CC terhadap massa lempung yang digunakan adalah 0%, 5%, 10%, 15%, 20%, dan 25%. Karakterisasi CC meliputi kadar abu dan karakterisasi CMCC meliputi uji porositas, analisis morfologi, dan distribusi unsur berdasarkan SEM-EDX. Hasil penelitian menunjukkan bahwa kadar abu CC sebelum dan setelah aktivasi adalah 5,625% dan 2,974%, berturut-turut. Hasil uji porositas CMCC menunjukkan bahwa semakin banyak karbon aktif tongkol jagung yang ditambahkan maka semakin besar porositas membran filter keramik. Komposisi unsur dalam CMCC didominasi oleh O, Si, Al dan C. Penambahan 10% karbon aktif tongkol jagung dapat menghasilkan komposisi optimum pada membran keramik. % penghilangan remazol red adalah 83,9%. Nilai COD limbah cair batik sebelum dan sesudah diolah dengan CMCC masing-masing sebesar 484,286 dan 26.339 mg/L.

Keywords: Ceramic membrane, Corncob, Activated carbon, Remazol red, Batik wastewater.

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# INTRODUCTION

One of the synthetic dyes that are often used as a batik dye is Remazol Red (RR). Remazol red has stable properties so that it does not fade quickly, but this remazol red dye liquid waste is carcinogenic. It can pollute the environment if it is not processed first. The environment is capable of naturally degrading

dyestuffs, but with limited capabilities, so it takes a very long time [1], [2], [3], [4].

Batik wastewater treatment has been carried out by various methods, such as electrolysis [5], coagulation [1], adsorption [6], and filtration [3]. However, each method has its advantages and disadvantages. The electrolysis method is carried out using direct electric current. The electrolysis method does not produce secondary waste and shorter processing time, but its

use depends on the specific electrode of the sample [7]. The coagulation method adds a coagulant or chemical substance to a solution to make suspended particles. The coagulation method is practical and easy to apply, but its use is influenced by pH and the number of colloid-forming particles [8]. The adsorption method uses an adsorbent that can absorb a particle. The adsorption method has a simple method, and the adsorbent that has been used can be regenerated, but the process is influenced by pH conditions and is difficult to use on a large scale [9].

In this study, the filtration method was chosen because it has more advantages over the previously mentioned methods. The working principle of the filtration method is to separate substances based on their particle size through a filter membrane. The advantages of the filtration method using a membrane filter are that it is not affected by temperature and pH, does not produce additional waste, is easy to combine with other processes, and the energy used for operation and maintenance is relatively low [3]. However, the filtration method also has drawbacks, namely the fouling or blockage caused by the accumulation of particles on the membrane surface. The filter membrane used is a clay-based ceramic filter membrane. The advantages of ceramic membranes lie in their excellent stability, resistance to chemical compounds and biological and microbial degradation, and relative ease of cleaning using cleaning agents [10].

The filtration process can be optimized by adding an adsorbent that functions as a particle absorber. The natural material that can be used as an adsorbent is corncobs. Corncobs can be used as adsorbents because they contain 41% cellulose, 36% hemicellulose, and 16% lignin [10]. Corncobs can be made into activated carbon to have a higher absorption capacity. Apart from being an adsorbent, the added activated carbon acts as a porosity agent to increase and enlarge the membrane pores. A filter membrane with a small porosity causes a low filtration rate, while a too-large porosity causes particle compounds to escape easily. This study used activated carbon from corncobs as an adsorbent and porosity agent. Corncobs were chosen because they are easy to obtain and cannot be utilized optimally. In addition, the use of corncobs is a form of agricultural waste management efforts. In this study, the effect of corncob-activated carbon on ceramic membranes on the porosity of ceramic membranes and its ability to

reduce the concentration of remazol red dye in batik wastewater is interesting to study.

# RESEARCH METODOLOGY

#### **Materials**

The chemicals used in this study were phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), remazol red dye, and distilled water from E. Merck. Batik liquid waste containing remazol red dye was taken from batik artisans in the Bantul area, corn cobs from agricultural waste, and kaolinite clay from Kasongan, Bantul Regency.

# Corncob-activated carbon (CC) preparation

The CC preparation steps were carried out according to the procedure reported by Rahayu [15] but with modifications. Corncobs were cut into small pieces and washed to remove impurities. Next is the dehydration process on the corncobs. dehydration process serves to remove the water content contained in the corncobs. The dehydration process is carried out by drying the corn cobs in the sun for 3-4 days until the corncobs are completely dry. The process of carbonization or composing is carried out by inserting corncobs into the furnace to be burned. Burning is carried out at 400°C for 4 hours in an airtight furnace. Corncobs that have become charcoal are removed from the furnace and cooled by leaving them in an open space. The corncob charcoal obtained was then ground into a fine powder with a size of 0.297 mm.

Furthermore, 100 g of corncob charcoal powder was activated into activated carbon by immersing it in a 15% H<sub>3</sub>PO<sub>4</sub> activator (200 mL) with an immersion time of 20 hours. After the soaking process, the activated carbon of the corncobs was filtered and then washed using distilled water until the pH became neutral. The corncob-activated carbon was then put into the oven at 120°C for 2 hours for the drying process, and the ash content was tested.

# Preparation of ceramic membrane modified with corncob-activated carbon (CMCC)

CMCC was made from 100 grams of kaolinite clay with 0.297 mm corncob-activated carbon as much as 0%, 5%, 10%, 15%, 20%, and 25% of the clay mass. Furthermore, each part is added with enough distilled water and mixed until homogeneous. Each mixture was then molded using a gypsum mold. The prints were then allowed to stand for 3 days. It was burned

at a temperature of 650°C-700°C for 8 hours to change the plastic properties of the clay to become stiff and strong. The morphology and elemental content of CMCC were characterized using Scanning Electron Microscope and Energy Dispersive X-Ray (SEM-EDX).

# **Porosity Test**

The CMCC porosity test was carried out by weighing the mass of each dry ceramic membrane. After that, the ceramic membrane was immersed in distilled water for 24 hours at room temperature and pressure. The wet ceramic membrane was then weighed. The data obtained is then calculated by equation (1) [12].

Porosity = 
$$\frac{P_2 - P_2}{P_1} \times 100\%$$
 (1)  
where :  
 $P_1$  = density of dry sample (g/cm<sup>3</sup>)  
 $P_2$  = density wet dry (g/cm<sup>3</sup>)

# Application of CMCC on batik liquid waste

Batik wastewater is diluted with distilled water with a volume ratio of 1:2. The batik wastewater that has been diluted is then put into each CMCC for the filtration process. After that, it was allowed to stand until the batik wastewater containing the remazol red dye came out through the pores of the CMCC. The results of the filtration process are stored in a container. The filtered solution that has been accommodated is then tested for the concentration of remazol red dye using a UV-Vis spectrophotometer and the COD (Chemical Oxygen Demand) value. A UV-Vis spectrophotometer was used to determine the concentration of remazol red in batik waste before and after being treated using a CMCC. Measurements were made at the maximum wavelength of remazol red, 511.5 nm. At the maximum wavelength, there is maximum absorption that can excite the electrons by a specific energy. The amount of remazol red successfully filtered by CMCC was determined from the difference in the concentration of remazol red in the batik waste before and after being filtered using a ceramic membrane. COD analysis was carried out at the Yogyakarta Institute of Technology Laboratory.

## RESULT AND DISCUSSION

#### **CMCC** characterization

Corncob charcoal activation was carried out using an H<sub>3</sub>PO<sub>4</sub> activator. Activation aims to remove water vapor and impurity compounds still present in the pores to increase the adsorption power. The determination of ash content aims to determine the content of metal oxides in activated carbon. Activated carbon made from natural ingredients not only contains carbon compounds but also contains several minerals. During the process of carbonization and activation, some minerals will be lost, and some will remain in the activated carbon [13]. The ash content test results of corncob charcoal are presented in Table 1. Corncob charcoal before activation had an ash content of 5.625%, while corncob charcoal that had been activated with H<sub>3</sub>PO<sub>4</sub> had an ash content of 2.974%. The ash content results of corncob production entirely meet SNI 06-3730-1995, namely the maximum ash content of 10%. Significant ash content can reduce the absorption of activated carbon, both for solutions and gases. The lower the ash content, the better the activated carbon ability. It is because the mineral content contained in activated carbon, such as potassium, calcium, sodium, and magnesium scattered on the activated carbon lattice, can cause the performance of activated carbon to decrease [14].

#### **CMCC Porosity Test**

The porosity test was carried out to determine whether or not pores were formed in the CMCC and to determine the percentage of the pore volume of the ceramic filter membrane [15]. The graph of the porosity test results for each ceramic filter membrane is shown in Figure 1. Based on Figure 1, it can be concluded that the more mass of CC added, the greater the porosity of the CMCC. It is shown that with every addition of 5% CC mass, the resulting porosity also increases, meaning that the addition of CC is directly proportional to the increase in porosity. The smallest porosity is CMCC, with an additional CC of 5%, 13.19%, and the largest is CMCC, with an additional CC of 25%, which is 49.52%.

Table 1. The results of the corncob ash content test and the quality standard of SNI 06-3730-1995

Treatment	Ash content (%)	
	Before Activation	After Activation
Single	5.670	2.950
Duplo	5.587	2.998
Average	5.625	2.974
SNI 06-3730-1995	Maximal 10	

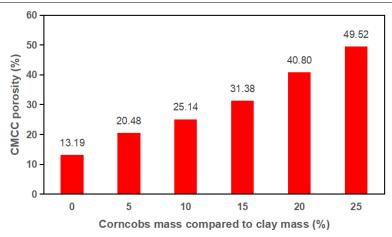


Figure 1. Corncobs mass relationship graph compared to clay mass (%) at CMCC to CMCC porosity

#### **CMCC Characterization Using SEM-EDX**

Surface morphology analysis and distribution of CMCC elements were characterized using SEM-EDX (Scanning Electron Microscope and Energy Dispersive X-Ray). The characterization of CMCC with SEM-EDX was carried out to determine the surface structure of the pores and the constituent elements contained in CMCC. The shape of the pore surface is one factor that plays a role in an adsorbent's ability to adsorb. The pores contained in CMCC can increase the adsorption ability because these pores are gaps that can expand the activated carbon surface [16].

The results of the CMCC analysis with the addition of 10% corncob-activated carbon using SEM are presented in Figure 2.

Based on the results of SEM images with a magnification of 1000 times and 5000 times in Figure 2, CMCC with an additional 10% CC has asymmetrical and irregular particle shapes. The structure's shape is caused by adding CC to the CMCC. The activation process can remove impurities on the activated carbon surface. The more impurities lost during the activation process cause the pore size of the activated carbon to increase [17].

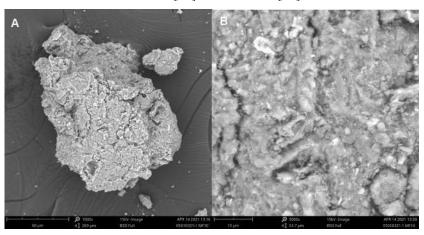


Figure 2. SEM results of ceramic filter membranes with the addition of 10% CC at magnifications (a) 1000 times and (b) 5000 times

The results of SEM images with a magnification of 5000 times were used for pore size analysis. Based on the analysis results, it is known that CMCC with 10% CC added has an average pore size of 8.5 m. The CMCC is a type of microfiltration where the microfiltration-type membrane has a pore size between 0.05 m - 10 m. Due to the asymmetrical and irregular shape of the particles, the ceramic filter membrane has a larger pore size due to the random arrangement of the particles. The resulting CMCC structure decreases the concentration of remazol red dye and the value of COD. It is due to the larger pore size causing the CMCC porosity to increase. CMCC with too small a porosity causes a low filtration rate, so it takes a very long time to pass through the membrane. However, if the porosity of the CMCC is too large, it can cause particles and organic compounds in the batik wastewater to escape quickly.

The constituent elements of CMCC were analyzed using EDX characterization. Samples fired with X-rays will reflect rays with a specific spectrum and wavelength that enter the detector. The signal that enters the detector will be converted into output in the form of absorption peaks based on each element. Ceramic filter membranes based on clay or clay have primary constituent elements such as silica and alumina. It is evidenced by the data from the EDX test results, displayed on the spectrum of the EDX test results in Figure 3.

EDX test result showed that the constituent elements of CMCC consist of oxygen (O) as much as 52.90%, silica (Si) as much as 17.11%, and aluminum (Al) as much as 14.04%. The oxygen content, which is quite large, is the oxide of the silica and alumina groups. It follows the composition of the clay compound, which is composed of silica and alumina

compounds [18]. Meanwhile, 8.68% of carbon (C) elements mainly come from CC, which is added to CMCC.

# Application of CMCC on batik liquid waste

The decolorization or decrease of the dye concentration is carried out by filtration using a ceramic filter membrane with the addition of corncob-activated carbon. Batik waste water containing remazol red dve is diluted first using distilled water. The dilution is done so that the batik waste water is easier to apply to the CMCC. In addition to containing dyes, batik waste water is also known to contain suspended solids from waxes used to form batik patterns and soda ash which functions as a colorlocking agent. Waxes and soda ash have a large enough molecular size to be retained in the pores of CMCC, while remazol red dve can be adsorbed by CC contained in CMCC. The decrease in remazol red dye concentration and COD values can occur due to the filtration and adsorption process at CMCC, which causes remazol red dye molecules and organic compounds to be retained on the CMCC surface [3]. The results of the reduction in the concentration of remazol red dye are presented in Figure 3. Based on Figure 3, each CMCC composition resulted in different efficiency values for reducing the concentration of remazol red dye. It is due to the variation of CC mass added in CMCC. CMCC with additional CC of 5%, 10%, 15%, 20%, and 25% had higher reduction efficiency values than CMCC without additional CC. However, with the addition of 15%, 20%, and 25% CC, there was a decrease in the ability of CMCC to reduce the concentration of remazol red dye, although it was not significant.

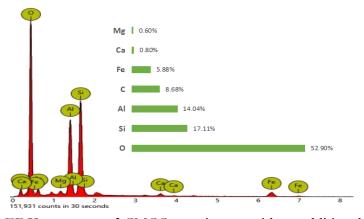


Figure 3. EDX spectrum of CMCC constituents with an additional 10% CC

The ability of CMCC decreased can occur because the adsorption capacity has been saturated and reaches equilibrium with the increased mass of activated carbon, which acts as an adsorbent so that the ability of CMCC decreases [19]. In addition, the porosity of CMCC also affects the ability of CMCC to reduce the concentration of remazol red dye. The more activated carbon added to the CMCC, the greater the porosity of the membrane [15]. The greater the porosity of the CMCC, the smaller the density of the CMCC. The smaller the density indicates the greater the number of pores and the wide pore size [20]. If the porosity of the CMCC is too large, the remazol red dye molecules and other organic compounds in the batik liquid waste can pass through the CMCC.

The mechanism of decolorization of remazol red dye can occur due to physical interactions and chemical interactions. Physical interactions occur on the surface of the CMCC. Remazol red dye molecules and organic compounds are larger than the pores of the CMCC, so these molecules are retained on the CMCC [3]. At the same time, the chemical interaction that occurs is the exchange of ions between activated carbon and dyes. Remazol red is a reactive dye with the molecular formula C<sub>27</sub>H<sub>18</sub>O<sub>16</sub>N<sub>7</sub>S<sub>5</sub>Na<sub>4</sub>Cl, which will dissociate to form an anion in the form of a sulfonate group (-SO<sub>3</sub>) when it becomes a solution. While on activated carbon, there is a hydroxyl group that undergoes protonation and forms H<sub>3</sub>O<sup>+</sup> on the surface of the adsorbent, next will interact with the sulfonate group (-SO<sub>3</sub>-) and cause the anion of the sulfonate group to be attracted to the surface of the adsorbent [21]. The mechanism that occurs is illustrated in Figure 4.

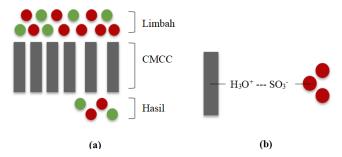


Figure 4. Mechanism of filtration of remazol red (red) dye and organic compounds (green) with CMCC (black) based on (a) physical interaction and (b) chemical interaction

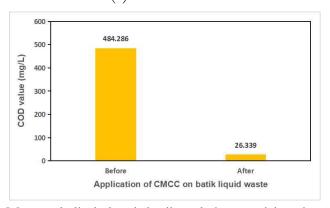


Figure 5. Penurunan nilai COD pada limbah cair batik pada komposisi optimum membran filter keramik

The batik wastewater filtration results at the optimum CMCC were then tested for COD. This test is carried out because the COD value is one of the parameters determining the quality of the waste. Waste with a high COD value indicates the level of pollution that occurs. Most pollutants in batik liquid waste come from dyes because only some of the dye is absorbed by textile materials, and the remaining 20-

50% is wasted as liquid waste [22],[23]. The COD value in batik wastewater will also decrease with the decrease in dye concentration after the filtration process [24]. The COD value decreased due to the reduced content of other organic and inorganic compounds, such as waxes, soda ash, and heavy metals, that were retained in the CMCC. The result of the COD value diagram is shown in Figure 5. Based on

Figure 5, the COD value in batik wastewater decreased after the filtration process. The COD value of batik wastewater which was originally 484.28% mg/L, can be reduced to 26.339 mg/L. CMCC, with the addition of 10% CC, reduced the COD value by 94.56%. The COD test results obtained have met the quality standards stipulated in the Regulation of the State Minister of the Environment No. 01 of 2010 the Implementation Water concerning of Environment Control, where the COD quality standard for the textile industry is 150 mg/L.

# **CONCLUSION**

Activated carbon from corncobs has been successfully applied to batik wastewater. Ceramic membranes with activated carbon from corncobs can reduce the dye content of batik liquid waste higher than active membranes without adding activated carbon from corncobs. The COD value of batik liquid waste after being processed using CMCC decreased significantly. It means that the performance of CMCC is very good for reducing the content of remazol red dye and the value of COD in batik wastewater.

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