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ADSORPTION OF LEAD (Pb) IN BATIK INDUSTRIAL WASTEWATER USING CELLULOSE-BASED ADSORBENT: A LITERATURE REVIEW

Sukmaningrum Latifah Oktaviani and Nurma Yunita Indriyanti*

Chemistry Education Study Program , Faculty of Teaching and Training,Universitas Sebelas, Surakarta, Indonesia

*correspondence, email: nurma.indriyanti@staff.uns.ac.id

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ABSTRACT

This literature review aims to determine the characteristics of the batik industrial wastewater, the type of adsorbent activation method most widely used to adsorb lead (Pb) in the batik industry wastewater, and the correlation between the source of cellulose and parameters on the adsorption ability of lead metal (Pb) with variations in adsorbent mass, pH and contact time in batik industrial wastewater. This literature review was carried out in 7 steps: exploring topics, searching, storing, and organizing information, selecting the required information, expanding the search, analyzing, and evaluating. Information and present the results. This literature review shows that Batik industrial wastewater contains BOD, COD, TSS, and heavy metals. The literature review obtained that the BOD and COD values came from the batik industrial wastewater of Jetis Sidoharjo with a value of 1775.5 mg/L and 16654.80 mg/L, the largest TSS value came from the batik industrial wastewater Gedhog with a value of 449 mg/L. The largest metal content of lead came from the batik industrial wastewater of Wiradesa, with a value of 7.654 mg/L. The most widely used activation method for treating adsorbents is the chemical activation method with strong acids such as HCl, HNO_3, and H_2SO_4. There is a correlation between the source of cellulose and parameters in the adsorption of lead metal in batik industry wastewater. Different sources of cellulose and parameters resulted in different adsorption capacities. Based on the literature review, the highest percentage of cellulose was found in sawn teak (60%), corn cobs (41%), rice straw (37.71%), rice husks (34%), and kapok seeds (21.83%). Maximum adsorption lies in the adsorbent with a mass of 0.1 – 1g, pH 5-7, and 30-45 minutes contact time.

Keywords: *Adsorbent, Cellulose, Batik Industry Wastewater, Lead (Pb)*

INTRODUCTION

The increase in the batik industry in Indonesia is in line with the increase in the liquid waste of the batik industry. The most batik-producing areas on the island of Java are spread over Central Java, West Java, East Java, Yogyakarta, Banten, and Jakarta, while outside Java, the largest batik industry is in the province of Jambi[1]. Waste is the result of residual waste generated from a production process, both industrial and domestic (household), better known as waste [2].

The increase in the batik industry will cause the wastewater load to be even greater. The wastewater generated in

Indonesia in 2017 was 883 tons/day, of which 29% of the load came from the textile industry[3]. The textile industry usually produces liquid waste thick in color and contains high and fluctuating BOD (Biochemical Oxygen Demand), COD (Chemical Oxygen Demand), pH, temperature, turbidity, salinity, and toxic chemicals. Sources of heavy metals chromium (Cr) and lead (Pb) which are toxic, can come from dyes ($CrCl_3$, $K_2Cr_2O_7$) or from mordant, which is a binder for dyes including $Cr(NO_3)_2$ and $PbCrO_4$[4], other sources of heavy metals such as Cu come from premet dyes and acids [5]

The increasing amount of batik industrial waste causes environmental pollution. One of the heavy metals in the batik industry wastewater is lead metal. Lead is one of the heavy metals that have high toxicity and is non-biodegradable so that its presence persists in nature lead, or what is known as lead, is symbolized by Pb having atomic number 82, the presence of lead in the human body can cause neurological disorders [6]

Suppose the batik industry liquid waste generated from each batik production process is directly discharged into the waters without being processed first. In that case, it will cause changes in the physical and chemical characteristics of the water and can threaten aquatic ecosystems. For this reason, waste needs to be processed first to comply with wastewater quality standards with parameters BOD, COD, TSS, and heavy metals. The maximum limit is BOD (60 mg/L), COD (150 mg/L), TSS (50 mg/L) and heavy metals (Pb; 0.1 mg/L) [7]. One of the waste treatments that can be applied is adsorption.

Adsorption was chosen because the required cost is relatively cheap, and the process is relatively simple, efficient, and highly effective [8]. One of the compounds that act as an absorbent or adsorbent is cellulose, found in various natural materials. Cellulose is a compound with functional groups that can bind with metal ions; the functional groups are carboxyl and hydroxyl groups [9]. A literature review related to the batik industry wastewater is needed to map and determine the characteristics of the batik industry wastewater, the most frequently used activation method, and to determine the relationship between cellulose sources with different parameters on lead adsorption.

METHODS

This literature review method consists of seven steps: exploring beliefs and topics, initiating the search, storing and organizing information, selecting or deselecting information, expanding the search, analyzing and providing information, and presenting the literature review results [10]. On the stage Exploring Beliefs and Topics, the reviewer searches for a topic that lacks knowledge about adsorbent. Initiating the search shows that literature searching was in the Google Scholar database used several keywords by using Boolean operators such as AND, OR, and Not. The Indonesian keywords used: Adsorbsi DAN limbah cair batik DAN Timbal DAN adsorbent cellulose; reviewer also used English keywords: Adsorption AND batik wastewater AND Timbal AND cellulose adsorbent. Next stage is storing and organizing information; the articles have been collected and saved into Mendeley software to make it easier to organize information. After that, the reviewer does select or deselect information, such as involves establishing criteria for determining which sources to use or not to use in this literature review. Criteria in selecting literature consist of searching for results based on keywords on the google scholar database (n = 251), selecting based on the 2015-2020 publication year (n = 151), selecting the relevant article (n=83), performing full-text screening (n= 39). The next stage is expanding the search, reviewer looking for additional information used by keywords Chemical Activation OR Physical Activation OR Physical-Chemical Activation (n = 10).

Six steps in a literature review are analyzing and providing information; in this stage, reviewers write down what data is obtained from the literature. The reviewer writes the data in 3 types of tables, the table among cellulose table and its parameters, the batik waste characterization table, and the activation method table. Finally, the data obtained will be

RESULTS AND DISCUSSION

Based on the literature review results, it was obtained the results of a review of the characteristics of the batik industry wastewater, the most frequently used activation method, and the source of cellulose and its parameter.

Characteristics of industrial batik wastewater

COD, BOD, TSS, and heavy metals can be found in the wastewater of the batik industry. BOD (Biochemical Oxygen Demand) is a characteristic that indicates the amount of dissolved oxygen required by microorganisms (usually bacteria) to decompose or decompose organic matter. Therefore, BOD can be interpreted as a description of organic matter easily decomposed in waters. At the same time, COD (Chemical Oxygen Demand) is the amount of oxygen needed to break down all organic matter contained in water. TSS (Total Suspended Solid) is a suspended solid that causes water turbidity, does not dissolve, and cannot settle [11]. Heavy metals in the batik industry wastewater include lead metal and chromium metal. The liquid waste content of the batik industry is different from one another. In the batik production process, chemicals, especially in dying or coloring, pelorodan, and washing. Chemicals used in the batik-making process include dyes as the main chemical and auxiliary chemicals, namely caustic soda (NaOH), soda ash (Na_2CO_3), baking soda ($NaHCO_3$), sulfuric acid (H_2SO_4), sulfites, and nitrites[4].

Besides being caused by the chemicals used, the content of the batik industry wastewater also depends on the amount of batik production and the presence of a wastewater treatment plant (WWTP). WWTP in the batik industry wastewater treatment process serves as a place to treat wastewater before being discharged into the waters. Based on the regulation of the

Minister of the Environment, the quality standard of batik wastewater for the content of BOD, COD, TSS, lead, and chromium is 60, 150, 50, 0,1 and 1 mg/L[7].

The liquid waste content of the batik industry in the Banaran Sukoharjo area is 615 mg/L COD, and the color intensity is 7000 PCU (Platina Cobalt Unit). Using dyes, this batik industry wastewater sample was taken from the batik dyeing process [3]. In the batik industry wastewater, there are 350 mg/L BOD, COD of 424 mg/L, and TSS of 375 mg/L. The wastewater sample used was taken from the UKM Batik Tulis Amali CH Sidoarjo with the condition that it had just come out of the rest of the batik coloring process[12]. One of the batik industries in Laweyan has COD and BOD values of 660.28 mg/L, and 1600 mg/L. This batik industry wastewater sample was taken at the WWTP location in the Laweyan batik village, which is located on Jalan Radjiman Laweyan, Surakarta City (behind the Laweyan village office) in an equalizing tank[13]. The liquid waste of the Yogyakarta Krebet batik industry has a COD value of 216.8 - 3845.5 mL. This wastewater sample from the Yogyakarta Krebet batik industry was obtained from each batik-making process[14]. The batik industry can produce liquid waste of 305 – 533 L/month and 7,712 m^3/month of solid waste. The following is a table of characteristics of the batik industry wastewater obtained based on a literature review:

Table 1. Characteristics Batik Industrial Wastewater

Sources	BOD (mg/L)	COD (mg/L)	TSS (mg/L)	Pb (mg/L)	Cr (mg/L)	Zat Warna	*Ref*
Laweyan Batik Industry	660,28	1600	-	-		-	[13]
Plenthong Yogyakarta Batik Industry	101,665	-	-	1,122		-	[15]
Banyuurip Ageng Batik Industry	-	-	-	3,015		-	[16]
Sokaraja Batik Industry	-	-	-	0,014		-	[17]
Krebet Batik Industry	-	216,8-3845,5	-	1-4	3,89	-	[14]
Jetis Sidoharjo Batik Industry	1775,5	16.654,8	208	-	<0,02	-	[18]
Jenes Batik Industry	-	-	-	0,215	1,517	-	[4]
Banaran Sukoharjo Batik Industry	-	615	-	-	-	7000 PCU	[3]
Batik Industry	-	-	-	0,252	-	-	[19]
Wiradesa Batik Industry	-	-	-	7,629	-	-	[20]
Tulis Batik Industry	-	9014,6	-	2,366	-	-	[21]
Gedhog Tuban Batik Industry	-	4951,75	448	-	7	-	[22]
Amali CH Batik Industry	350	424	375	-	-	-	[12]

According to 2014 environment ministerial regulation for BOD, COD, TSS, Pb, and Cr, the quality standard limit for textile wastewater content is 60; 150; 50; 0,1; 1 (mg/L). In the wastewater of the Jetis batik industry, there are BOD, COD, and TSS in the amount of 1775.5 mg/L, 16,654.80 mg/L, 208 mg/L. This batik industry wastewater sample was obtained from the batik coloring process. At the location of the Kampung Jetis batik industry, there is no WWTP due to the unavailability of sufficient land for the construction of the WWTP at Kampung Jetis Sidoarjo. Based on the research conducted, batik industrial wastewater contained COD of 9014.6 mg/L.

Based on Table 1, batik industrial wastewater contained BOD, COD, TSS, and heavy metals. The largest BOD value is in the batik industry wastewater from Jetis, Sidoharjo, with 1775.5 mg/L. The largest COD value is in liquid batik waste originating from Jetis, Sidoharjo, with 16654.80 mg/L. Meanwhile, the largest TSS was found in the batik industry wastewater, where COD was 4951.75 mg/L, and TSS was 448 mg/L. This liquid waste sample was obtained from one of the batik production sites in Tuban with the name batik Gedhog. The batik industry originates from the Tuban gedhog batik industry with 449 mg/L. The highest levels of lead are found in the liquid waste of the Wiradesa batik industry, with a level of 7.654 mg/L. While the largest concentration of chromium metal is in the batik industry wastewater on the banks of the Jenes Laweyan river at 1,516 mg/L. .

The difference in content in the batik industry wastewater is caused by chemicals, especially in dyeing or coloring and washing in the batik production process. Chemicals used in the batik-making process include dyestuffs as the main chemical and auxiliary chemicals, namely caustic soda (NaOH), soda ash (Na_2CO_3), baking soda ($NaHCO_3$), sulfuric acid (H_2SO_4), sulfites, and nitrites [4]. Apart from being caused by chemicals used, the content of the batik industry wastewater also depends on the amount of batik production and the presence of a wastewater treatment plant (WWTP). WWTP in the batik industry wastewater treatment process serves as a place to treat wastewater before it is discharged into the environment.

Activation Method On Adsorbent

Activation is a treatment of adsorbent that aims to enlarge the pores by breaking the hydrocarbon bonds or oxidizing the surface molecules so that the charcoal changes physical and chemical properties. The surface area increases and affects the adsorption power [23].

The interaction between the activating agent and the structure of the carbon atoms resulting from the carbonization is the mechanism of the activation process. Physical activation can be done using heat, steam, and CO_2 gas, while chemical activation uses activators [24]. Meanwhile, Physico-chemical activation is done by combining physical and chemical activation.

The activation of the adsorbent is divided into two types, namely hardwood, and softwood. Hardwoods contain more cellulose and extractives and less lignin than softwoods; For pulp purposes, softwood fibers are, on average, more than three times the length of those contained in hardwoods (thus, the origin of the terms “long-fiber pulp” and “short-fiber pulp”). Furthermore, since increasing fiber length usually translates into more inter-fiber bonding, softwood pulps impart greater strength to the products into which they are made than hardwood pulps manufactured by the same process[25].

Examples of hardwood adsorbents are rubber sawdust, pine sawdust, merbau wood sawdust, kapok seed, and cocoa husk. Meanwhile, softwood adsorbents such as rice husk, pomelo leaves, kepok's banana, jengkol skin, and dami jackfruit.

Table 2 shows activation methods for hardwood adsorbents consisting of rubber sawdust, pine sawdust, Merbau wood sawdust, kapok seed charcoal, and cocoa husks.

Table 2. Activated Methods On Hardwoods Adsorbent

Adsorbent	Activation Methods	Treatment	Result	Ref
Rubber Wood Sawdust	Physic-Chemistry	Heated in an oven at 110 ^{0} C for 24 hours, then soaked in 11 ml 98% H_2SO_4 and 6.6 ml 65% concentrated HNO_3.	Activated carbon with a size of 0.5 - 1 mm at a temperature of 110 ^{0}C, the average pore size of the adsorbent is 42.6 A, and the adsorption capacity is 38.56 mg/g	[26]
Pine sawdust	Chemistry	Activated with 0.1 N NaOH for 24 hours	Pine sawdust adsorbent with an efficiency 99,84%	[27]
Merbau Wood Sawdust	Chemistry	Activated with HCl 2N for 24 hours	Merbau wood sawdust adsorbent activated with capacity adsorption 1,925 mg/L.	[28]
Kapok seed charcoal	Chemistry	Soaked in H_3PO_4 85% for 24 hours	Kapok seed charcoal activated with capacity adsoprion 0.05 mg/g.	[29]
Cocoa husk	Physics	Cocoa husk carbonated with 600 ^{0}C for 15 minutes	Charcoal cocoa husk with percentage of adsorption 98,715%	[30]

Research using Rubber Wood Sawdust adsorbent using physicochemical activation method by heating at 110 ^{0}C in a 24-hour oven, then soaking in 11 ml 98% H_2SO_4 and 6.6 ml 65% HNO_3 obtained carbon with an active size of 0.5 – 1mm with an average pore size of 42.6 A for adsorbent and an adsorption capacity of 38.56 mg/g. Furthermore, sawdust was obtained with an adsorbent derived from pine sawdust chemically activated using 0.1 N NaOH for 24 hours with an adsorbent efficiency of 99.84%[26].

Activation using NaOH solution to cause delignification of the adsorbent; Delignification serves to dissolve compounds such as lignin so that it inhibits the adsorption process. Meanwhile, lignin will inhibit the ion transfer process to the active site of the adsorbent. In addition, NaOH solution will break the bonds of cellulose with lignin. The OH- ions from the

NaOH solution will break the bonds from the basic structure of the lignin. Figure 1 is a mechanism for breaking the bonds of cellulose derived from rice straw with lignin.

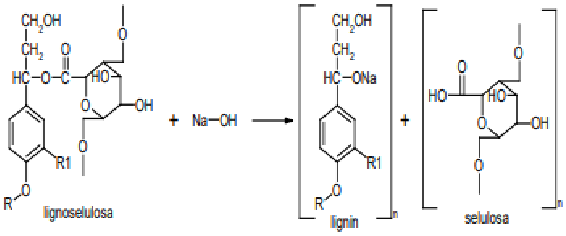

Figure 1. The mechanism of breaking the bond between lignin and cellulose by NaOH.

Lignin degradation begins with the attack of the H atom bound to the phenolic OH group by hydroxide ions (OH^-) from NaOH. The H atom in that section is acidic because it is bonded to an O atom with a large electronegativity. The more electronegative O atom will attract electrons to the H atom so that the H atom will be positively charged and easily released into H^+ ions. Acidity is also influenced by the resonance effect of the alkyl group at the para position so that the H atom in the phenolic group will be more acidic [31]

Research using adsorbent derived from kapok seed charcoal activated using chemical methods through immersion with 85% H_3PO_4 for 24 hours obtained kapok seed activated charcoal adsorbent with adsorption capacity of 0.05 mg/g. The charcoal activation process by immersion in 150 ml of 85% H3PO4 solution for 24 hours is intended to open the pores of the charcoal surface. This process works by breaking hydrocarbon bonds or oxidizing surface molecules. As a result, the activated charcoal of kapok seeds undergoes physical and chemical changes, namely increasing surface area and increasing the absorption of activated charcoal. For example, the following is an activation reaction of charcoal with H_3PO_4:

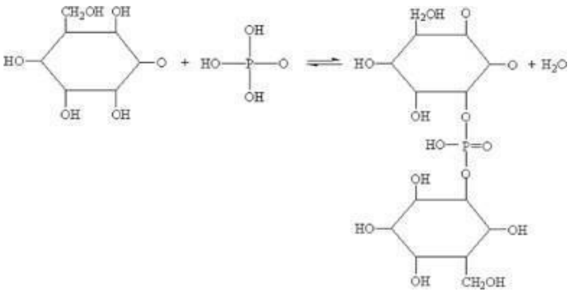

Figure 2. The activated reaction between charcoal and H_3PO_4

The figure shows that H_3PO_4 reacts with the formed charcoal and then forms micropores on the surface, which serve as a place for adsorption. The surface of the adsorption on the charcoal wider [32]

Activation methods on softwood adsorbents derived from rice husk, pomelo leaves, *Dami* jackfruit, *Kepok* banana peel, and *Jengkol* peel are divided into physical, chemical, and chemical Physico-chemical activation methods. For example, a study using an adsorbent derived from *Jengkol* skin was chemically activated through immersion in 5N nitric acid for 90 minutes and then sieved on a 100 mesh sieve to obtain an adsorbent of jengkol skin. Table 3 shows activation methods for softwood adsorbents:

Table 3. Activated Methods On Softwood Adsorbents

Adsorbent	Activated Methods	Treatment	Result	Ref
Rice Husk	Physic-Chemistry	Carbonized at 400 -600 ^{0}C for 90-150 minutes, soakan on HCl, filter with100 mesh sieve	Activated carbon with 4.86% humidity, 30.04% Ash, and 15.76% volatile matter	[33]
Pomelo Laves	Physics	Dried on oven at 65 ^{0}C and filter	Pomelo Leaves adsorbent with size <355μm	[34]
Dami Jacfruit	Physisc	Carbonized at 250 ^{0}C, 2.5 hours	Jackfruit dami charcoal with adsorbent absorption efficiency of 96.67%.	[35]
Kepok's Banana Peel	Chemistry	Soaked for 90 minutes in 5M concentrated nitric acid, sieved through a 100 mesh sieve	Activated kepok banana peel adsorbent with a size of 100 mesh	[36]
Jengkol Skin	Chemistry	Soaked in 5N nitric acid for 90 minutes, sieved on sieve 100 mesh	Jengkol peel adsorbent is activated with size 100 mesh	[37]

A study used an adsorbent derived from jengkol skin was chemically activated through immersion in 5N nitric acid for 90 minutes and then sieved on a 100 mesh sieve to obtain an activated jengkol skin adsorbent with a size of 100 mesh. Nitric acid is an activating agent because it has H^+ ions that can dissolve metal impurities by exchanging metal ions bound to the biosorbent. This process causes an increase in the number of active biosorbent sites that have H^+ ions. In addition, soaking with nitric acid also serves to break lignin from the lignocellulosic complex. Breaking lignin is necessary because lignin can cover the hydroxyl groups of cellulose in biosorbents by forming covalent bonds with lignin chromophores[37].

Based on Table 2 and Table 3, the activation method that is most widely used to activate the adsorbent is the chemical activation method. The most widely used compounds in this activation method are acidic in the form of strong acids such as nitric acid (HNO_3), hydrochloric acid (HCl), sulfuric acid (H_2SO_4), and phosphoric acid (H_2SO_4). This acid compound is widely used because it has H^+ ions which can dissolve ions that pollute the surface of the adsorbent. As a result, it is used to open the pores of the adsorbent surface by breaking hydrocarbon bonds or oxidizing surface molecules so that they undergo physical and chemical changes, and the surface area increases. Large and increase the adsorption capacity of the adsorbent.

Sources of Cellulose and Parameters

Cellulose is a polysaccharide with a very high relative molecular mass; cellulose comprises 2000 - 3000 glucose[38]. The existence of cellulose is very abundant in nature because it can be found on every part of the plant, such as roots, stems, and twigs. Cellulose is the main constituent of plant cell walls which is very abundant. Tabel 4 is a table source of cellulose and parameters.

Table 4. Sources of Cellulose and Parameters

Sources	Modes	Efficiency of Adsorption (%)	% Cellulose	[Pb] (ppm)	Variants Mass adsorbent (g)	Contact Time (minutes)	pH	Ref
Corncob raw	*batch*	97,29	41	58,86	0,02 ; 0,04 ; 0,06 ; 0,08 ; 0,12 **(0,08)**	-	-	[39]
Kapok seed	batch	97,76	21,83	118	0,3 ; 0,6 ; 0,9 ; 1,2 **(1,2)**	30	-	[29]
Rice husk	batch	88,765	37,71	5 ; 10 ; 15 ; 20 ; 25 **(10)**	0,5	30	4; 5; 6; 7; 9 **(5)**	[40]
Jati wood sawdust	batch	99,98	60	61,44	0,1; 0,2 ; 0,3 ; 0,4 ; 0,5 **(0,4)**	-	3 ; 4 ; 5 ;6 ; 7 ; 8 **(7)**	[41]
Agave baggase	batch	93,14	-	-	0,4	5-120 **(15)**	3-6.5 **(5.5)**	[42]
Mangifera Indica	batch	69	-	100	0,1	240	5	[43]
Kakao skin	batch	98,715	17,27	100	0; 0,5; 1; 1,5(**0,5)**	30; 40;50; 60; **(40)**		[30]

Based on research with corncob adsorbent raw material with 41% cellulose content, it has an adsorption efficiency of 97.29% to adsorb lead (Pb) with the most effective adsorbent mass of 80 mg. This study uses adsorbent weight variations, 20, 40, 60, and 80 mg. The weight of the adsorbent increased at 20, 60, 80, and 120 mg, causing an increase in the percentage of lead adsorbed. The weight of the adsorbent affects the adsorption process because as the weight of the adsorbent increases, the value of % Pb adsorbed to ions also increases and reaches equilibrium. However, the adsorbent weight of 40 mg decreased. The small lead absorption is due to the contact time between the adsorbate and adsorbent that exceeds the optimum time, which can cause desorption. Then, at 120 mg, the absorption decreased due to the saturated adsorbent.

Based on the description and Table 4, the five most significant percentages of cellulose are found in teak saws with a percentage of cellulose at 60%, corn cobs at 41%, rice straw at 37.71%, and rice husks at 34%, and kapok seeds 21.83%. Based on the mass range of the adsorbent, the maximum adsorption lies in the adsorbent with a mass range of 0.1 - 1 g. Total adsorbent affects the adsorption process. The increasing weight will cause the adsorbent to reach saturation if the surface has been filled with an adsorbate. Due to the desorption process, there will be a decrease in adsorption capacity after reaching the maximum value.

Based on the pH range in Table 4, the pH range for maximum adsorption of batik liquid waste lies at pH 5-7. At high pH, precipitation of Pb ions occurs, which reduces the solubility of Pb ions in solution; this causes a decrease in the number of Pb ions that the cell surface can absorb. In addition, the high pH causes the presence of more OH^- ions so that metal ions begin to precipitate. Again, this causes the absorption to be reduced [41]. Absorption is also low if the pH occurs below the optimum range because the H^+ concentration is too high; it will be able to block the interaction between metal ions and the adsorbent.

Based on the contact time in Table 4 to adsorb lead metal, the maximum results are 30-45 minutes. Although the adsorption of ions from a solute will increase if the contact time is longer, the time to reach an equilibrium state in the metal adsorption process by the adsorbent ranges from several minutes to several hours. Suppose the adsorbent is contacted in a relatively short period. It will result in not many functional groups of the adsorbent interacting with the metal in the solution. There are not many functional groups that play a role in the adsorption of lead metal.

CONCLUSION

Batik industrial wastewater contains BOD, COD, TSS, and heavy metals. The largest BOD and COD values are in the batik industry wastewater from Jetis, Sidoharjo, with 1775.5 mg/L and 16654.80 mg/L. The largest TSS is found in the liquid waste of the Gedhog Tuban batik industry with the amount of 449 mg/L. The highest levels of lead are found in the liquid waste of the Wiradesa batik industry, with a concentration of 7 ppm. The largest concentration of chromium metal is found in the wastewater of the batik industry, which is on the banks of the Jenes Laweyan river, with an amount of 1.516 mg/L. The most widely used activation method for treating adsorbents is the chemical activation method using acidic compounds in strong acids, HCl, HNO_3, and H_2SO_4. There is a relationship between the source of cellulose and its parameters in the adsorption process of lead metal in the batik industry wastewater; different sources of cellulose and parameters will result in different adsorption capacities in the adsorption of lead metal.

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