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Treatment of Laboratory Wastewater by Using Fenton Reagent and Combination of Coagulation-Adsorption as Pretreatment

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ABSTRACT

Laboratory wastewater contains organic and inorganic compounds that are harmful to the environment when disposed of without prior treatment. Besides the high COD and BOD values, the laboratory wastewater also contains metals such as iron (Fe), zinc (Zn), copper (Cu), chromium (Cr), and lead (Pb) which is categorized as dangerous waste material and can pollute the groundwater. Although the quantity of wastewater produced by the laboratory is relatively small, it has a real impact on the environment around the laboratory. However, the wastewater has to be treated properly before being discharged into the environment. The aim of the research was to study the laboratory wastewater treatment by using Fenton's reagent with coagulation and adsorption pretreatment. In the pretreatment with coagulation, three types of coagulants are used, namely PAC (Poly Aluminum Chloride), ACH (Aluminum Chlorohydrate) and Aluminum Sulfate (AS) with their respective concentrations of 10–80 ppm. The highest percentage of average pollutant removal of 58.21% was found when 80 ppm of AS was applied. The pretreatment was continued by adsorption with activated carbon and zeolite adsorbents within 60–120 minutes of mixing time. It was detected that the most optimum adsorbent was activated carbon with average pollutant removal of 50.22% within 1 hour of mixing time. Processing was extended by utilized Fenton's reagent using a variation of the molar ratio between 1:100 and 1:400. It was obtained that the best molar ratio to degrade the laboratory wastewater is 1:300 with an average removal of pollutant of 43.45%. As a result of laboratory wastewater treatment using combine Fenton's reagent and coagulation-adsorption pretreatment, an average pollutant removal of 90.81% was obtained. The final content of COD, BOD, TSS, as well as Cu and Pb metal has met the environmental quality standard.

Keywords: laboratory wastewater, fenton reagent, coagulation-adsorption, heavy metal.

INTRODUCTION

Laboratory wastewater is the result of chemical residues used during student's practicum and research. Although the quantity of wastewater produced by the laboratory is relatively small, it has a real impact on the environment around the laboratory. Laboratory wastewater can contain metals such as iron (Fe), zinc (Zn), copper (Cu), chromium (Cr), and lead (Pb), depending on the module of the student's practicum. Heavy metal waste is very dangerous for the sustainability of the environment and the organisms involved, including humans. Heavy metal waste cannot be degraded by microorganisms, so that if the waste is not treated immediately, the waste will accumulate in the environment and cause pollution. On the basis of the data from the research conducted by (Audina et al., 2016), it was stated that Environmental Engineering Laboratory wastewater has a COD content of 611.4 mg/L and a metal content of 19.40 mg/L of Fe, and 22.90 of mg/L Pb. It shows that the heavy metal levels in the laboratory are very dangerous, because they exceed the permissible threshold. The maximum standard concentration of wastewater contamination set by the Ministry of Environment of the Republic of Indonesia (RI) for Fe and Pb are 5 mg/L and 0.1 mg/L, respectively.

Laboratory wastewater, in addition to containing heavy metals, also contains COD, BOD, TSS, TDS and an uncertain pH, so it must be treated using coagulation or adsorption methods, or other methods, usually a combined method is also required in managing the wastewater. On the basis of the results of previous tests and studies, it was known that to treat laboratory wastewater that meets the quality standard set by the Ministry of Environment of RI, a pretreatment and treatment process is required in accordance with the substance contained in the wastewater. In this study, the laboratory wastewater treatment is carried out using coagulation and adsorption pretreatment. The coagulation process is substantially effective in removing high molecular weight organics. Coagulation can remove contaminants of different origins, and it is most commonly used most approach (Kyrii et al., 2020). This method is simple, allows the usage of different coagulants. In addition, the simultaneous use of coagulation and adsorption process can also be effective in removing organic substances (Dąbrowska, 2021).

Fenton's reagent method is used because it has a short reaction time. Other advantages are that the process is easy to carry out and control, iron and H_2O_2 are cheap and non-toxic, mass transfer limitations do not exist because the catalyst is homogeneous, and there is no energy involved as a catalyst. Fenton's reagent has a function as a degrading contaminant that is difficult to decompose in an effluent to reduce the levels of Chemical Oxygen Demands (COD) in laboratory wastewater. Pretreatment in the test was carried out to remove Total Suspended Solids (TSS), pH, and heavy metals.

Audina et al. in 2016 used a combination process of coagulation and adsorption, the parameter values tested still did not meet the quality standards of wastewater pollution according to Ministerial Regulation No. 5 of 2014 (Audina et al., 2016). Hence, it is necessary to combine coagulation-adsorption methods with another method such as Fenton's reagent method. Fenton reagent is very effective for treating wastewater the COD concentration of which is in the range of 500 mg/L using a molar ratio of 1:300 mmol (Cahyana & Pemadi, 2018). Another research was conducted by Dung et al., leachate was treated by a combination of Fenton and coagulation processes. The result was the effluent can meet the treatment requirement by the discharge standards (Dung et al., 2020).

This study aimed to examine the laboratory wastewater treatment by using Fenton's reagents with coagulation and adsorption method as pretreatment. The effect of coagulants type and coagulants concentration, the adsorbent type, and Fenton's reagent molar ratio, on the pollutant removal were also studied.

MATERIALS AND METHODS

Materials

The materials used are aquadest, Poly Aluminum Chloride (PAC), Aluminum Chlorohydrate (ACH), Aluminum Sulfate (AS), zeolite, Sodium Hydroxide, Sodium Thiosulfate ($Na_2S_2O_3.5H_2O$), Hydrogen Peroxide (H_2O_2), $FeSO_4.7H_2O$, and activated carbon were obtained from Merck. The wastewater sample was taken from Pharmacy Laboratory in Universitas Sriwijaya.

Methods

Coagulation

In preparation for the initial testing of the original wastewater from the Pharmacy Laboratory, the parameters of BOD, COD, TSS, pH, Fe, Zn, Cu, Cr Total and Pb, were first tested. After the initial testing of the wastewater sample was carried out, it was followed by the coagulation method.

First, 500 mL of the sample was placed into a 1 L beaker glass, coagulant of ACH, PAC, and AS was prepare, each with the concentration of 10, 20, 40, 60, and 80 ppm. Stirring was conducted by using fast rotation of 150 rpm for 10 minutes, then continued by a slow stirring of 30 rpm for 10 minutes. The decreased in BOD, COD, pH, TSS, Fe, Zn, Cu, total Cr, and Pb were analyzed (Teguh et al., 2022).

Activated carbon and zeolite activation

The synthetic zeolite was activated by smoothing the zeolite with a mortar; then, it was sieved with a 400-mesh sieve. Afterwards, the zeolite was washed using distilled water while stirring repeatedly. The washed zeolite was filtered and dried using an oven at 110°C for 2 hours (Subariyah et al., 2013). The activated carbon was soaked using 15% of NaOH for 24 hours, then filtered with a filter paper. Activated carbon that was filtered was placed into a furnace at a temperature of 500°C for 2 hours (Maulana et al., 2017).

Adsorption

Initially, 100 mL of waste was placed into a 1 L beaker glass; then, 1 g of activated carbon was added into the beaker. Stirring was carried out for 1 and 2 hours using an overhead stirrer at 200 rpm and then filtered using a filter paper. The decreases in BOD, COD, pH, TSS, Fe, Zn, Cu, total Cr, and Pb were analyzed. The procedure was repeated using 1 g of synthetic zeolite (Febrina et al., 2019).

Fenton process

First, 150 mL of laboratory wastewater sample was poured into a beaker glass. The pH was adjusted to 3 by adding 0.1 M NaOH or 0.1 M H_2SO_4. Fenton's reagent was added with the ratio molar of Fe and H_2O_2 solution being 1:100. The solution was stirred for 1 hour with a magnetic stirrer. After stirring for 1 hour, 0.5 ml $Na_2S_2O_3.5H_2O$ 1 N was added to stop the reaction. The solution was filtered to separate the precipitate using a filter paper. The same steps were performed for Fenton's reagent ratios of 1:200; 1:300, and 1:400. The decreases of BOD, COD, pH, TSS, Fe, Zn, Cu, total Cr, and Pb were analyzed for each solution (Agustina et al., 2016).

Analysis of removal percentage

The analysis of the COD, BOD, TSS, Zn, Cu, Fe, Cr total, Fe and Pb levels was carried out by requiring the final concentration of each substance. The removal percentage (η) of the substance is calculated by the following equation:

$$\eta = \frac{Co-C}{Co} \text{ x } 100\% \quad (1)$$

where: *Co* is the initial concentration before each treatment, *C* is the final concentration after the sample was treated.

The results obtained are compared to the wastewater quality standards according to the Regulation of the Ministry of Environment of RI No. 5 of 2014.

RESULTS AND DISCUSSION

Effect of coagulant type and coagulant concentration on pollutants removal

The pH is an indicator to determine the level of acidity or alkalinity of a solution. The pH is very important in coagulation process. When positively charged ions in the coagulant encounter negative ions at certain pH conditions, particles or flocs are formed. The particles will continue to increase in size and weight so that the floc tends to settle. The process of floc formation causes the pH value to tend to decrease.

The coagulation process uses three types of coagulants, i.e. PAC, ACH, and AS, to treat the laboratory wastewater. The coagulation process aims to destabilize colloidal particles in water. The coagulation process is carried out by considering various factors, such as stirring speed, stirring time, and coagulant dose.

The effect of increasing the concentration of ACH, PAC, and AS coagulant on the pH value can be seen in Figure 1. ACH is the most concentrated dissolved aluminum based coagulant available and has the highest option. After the addition of 10 ppm ACH coagulant, the pH value decrease, but the pH increases after adding the higher concentration of the ACH. The decrease in the pH value is due to the formation of acidic compounds formed from hydrogen ions. Meanwhile, the increase in the pH value after the addition of coagulant occurred because the excess aluminum hydroxide compound was split into Al^{3+} and $3OH^-$ hydroxide compounds, which in excess caused the pH of the water rise. Aluminum hydroxide has amphoteric properties that enable it to act as an acid or a base in a solution.

In the coagulation process using PAC coagulant, the floc formed was faster than ordinary coagulants. This is due to the aluminate active group that works effectively in binding colloids, where this bond is strengthened by polymer chains from the polyelectrolyte group, so that the floc becomes denser.

The addition of hydroxyl groups to the hydrophobic colloidal chain will increase the molecular weight. After the addition of PAC coagulant, the pH value increased as the concentration of PAC increased. The increase in pH value is due to the absence of free hydrogen ions (H^+), resulting from the hydrolysis reaction when the coagulant reacts with water.

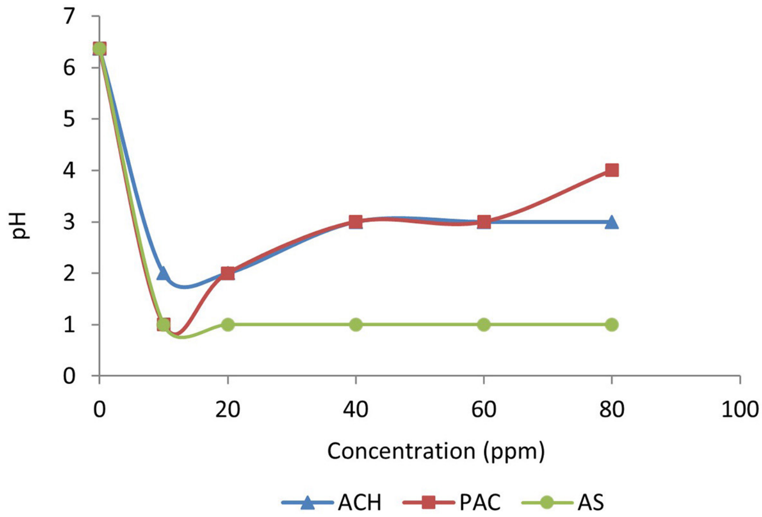

Figure 1. Effect of ACH, PAC, and AS coagulant concentration on pH values

However, the use of AS coagulant causes the release of hydrogen for each hydrogen group. The hydrogen ions produced will cause a decrease in pH so that the treated water becomes more acidic. The addition of AS tends to lower the pH of the solution because AS produces H^+ ions after reacting with water. The decrease in pH that occurs also because of AS will produce H_2SO_4 compounds when dissolved in water, which reduce the pH value of solution (Aziz et al., 2013). The pH of the wastewater sample was still low after increasing the concentration of AS, as it can be seen in Figure 1.

Chemical oxygen demand

Chemical oxygen demand (COD) is the amount of oxygen needed to chemically oxidize organic matter in water. COD is important to determine the approximate amount of oxygen that will be required to stabilize the biologically present organic matter and to determine its compliance with the permissible limits for wastewater discharge.

Figure 2 shows the COD reduction in the wastewater sample using different concentrations of ACH, PAC, and AS coagulant. The lowest COD value of 309 mg/L was reached when using 10 ppm of AS. The AS ($Al_2(SO_4)_3$) can reduce suspended substances contained in the wastewater. This is because the Al cations in AS will undergo a hydrolysis reaction to form dissolved Al species or aluminum hydroxide precipitates.

The dissolved Al formed is in the form of monomers and some of them are positively charged which can neutralize negatively charged colloidal particles. Negative organic content tends to be adsorbed and trapped in

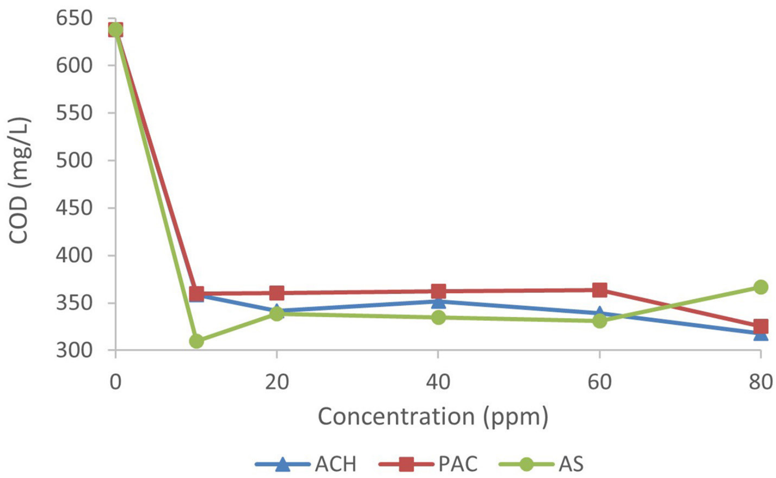

Figure 2. Effect of coagulant concentration on COD reduction

aluminum hydroxide deposits (Sutapa, 2014). This causes the COD value in the waste decrease.

Total suspended solid

Total suspended solid (TSS) measurement was carried out because this parameter can cause increased turbidity in the water. Laboratory wastewater has a TSS content of 88.8 mg/L. This study varied the ACH, PAC, and AS coagulants with variations of each coagulant between 10–80 ppm with the aim of obtaining the optimum dose to eliminate TSS. The removal of TSS against coagulant variation and dose variation has been also explored. There was a decrease of up to 93.24%.

The dose of coagulant has a great influence on the success of coagulation, giving a small dose causes the floc formation process to lack a floc nucleus so that it leaves more colloidal particles. With the increasing dose given, the colloid particles that combine to form macro floc are more and more and leave less colloid. At 40 ppm ACH coagulant, there was a significant increase in the amount of TSS. This happens because at that concentration the ACH given exceeds the optimum limit, which results in delays in the floc formation process, because too many cations result in electrostatic forces on the colloid which are already fused on the surface.

The macro floc becomes large and results in the destruction of the bonds that have been formed. The increase in TSS levels is caused by the stabilization of colloid particles due to excessive doses. Destabilization is a process of reversing the charge of colloidal particles which is generally almost all colloidal particles in the water are negatively charged and turn positive due to absorption of the excess dose which results in repulsion between colloidal particles because they have the same charge so they cannot form larger flocs and cause an increase in the TSS levels in the sample again (Ainurrofiq et al., 2017).

Biological oxygen demand

BOD (Biological oxygen demand) is an approximate measure of the amount of biochemical that is degraded in the waters. BOD is an oxygen requirement for a number of bacteria to decompose or oxidize organic substances dissolved or suspended in water into simpler organic materials (Siregar, 2005). The high BOD value is influenced by the type of wastewater, the degree of acidity (pH) and the higher content of organic compounds. On the basis of the initial testing of the wastewater, the BOD value was 97.3 mg/L which meets the quality standard.

The decrease in BOD concentration was influenced by the coagulant dose factor. The coagulant can bind or absorb suspended or organic particles so that the particles are successfully precipitated. The decrease in the number of suspended particles in the laboratory wastewater will decrease the BOD (Siregar, 2005). The highest BOD reduction of 91.78% was found when using 80 ppm of AS coagulant, as it can be seen in Figure 3.

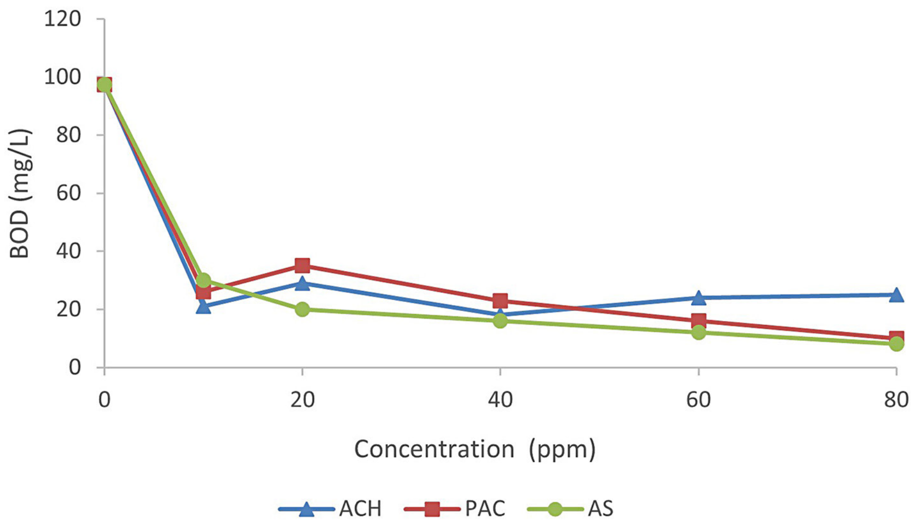

Figure 3. Effect of coagulant concentration on BOD reduction

Heavy metal reduction

Copper (Cu)

Laboratory wastewater has a Cu content of 19.6 mg/L. This study varied the coagulants of ACH, PAC, and AS with variations of each coagulant concentration of 10–80 ppm to obtain the optimum dose to remove the total Cu. Figure 4 shows that the optimum Cu reduction occurred by using 80 ppm of PAC coagulant. In this concentration, the solution has the highest pH, so the Cu metal experienced a great decrease to 4.43 mg/L. The higher the pH, the greater the percentage of reduction in Cu metal in the wastewater (Nurhasni et al., 2013). This is due to the interaction between the hydrogen ion and the Cu ion.

Chromium (Total of Cr)

The wastewater containing chromium metal is included in the category of hazardous and dangerous waste material. Chromium is a metal that is difficult to degrade, so it can last a long time in water. Laboratory wastewater has a total of Cr content of 70.2 mg/L. This study varied the type of ACH, PAC, and AS coagulants and the concentration of each coagulant (10–80 ppm) to obtain the optimum dose to remove total Cr.

Figure 5 demonstrates the most optimum reduction of total of Cr occurred when using 80 ppm of ACH coagulant, to the level of 52.78 ppm. The Cr metal deposit is in the form of colloids and small flocs which will be bound to form large flocs with the addition of coagulant.

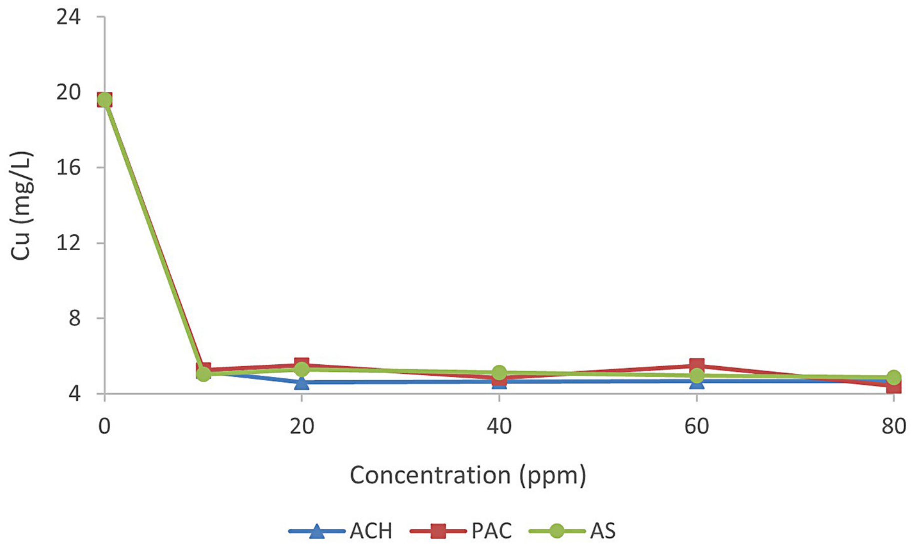

Figure 4. Effect of coagulant concentration on Cu reduction

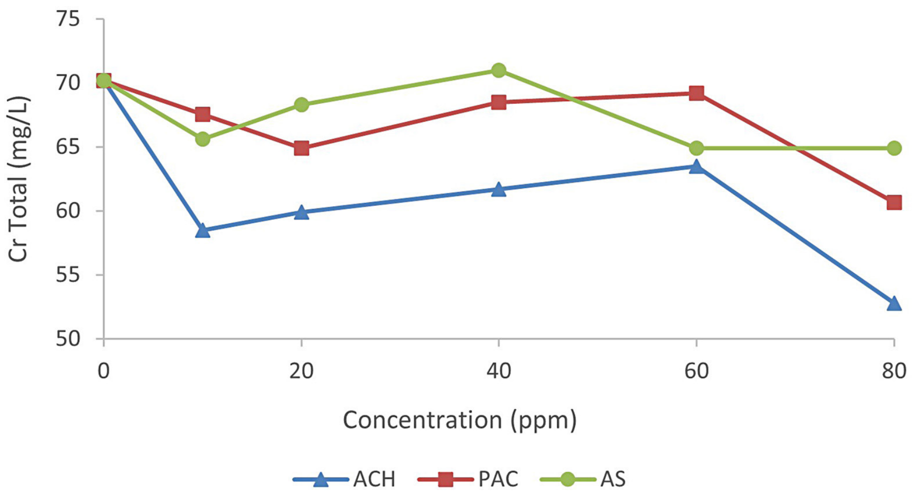

Figure 5. Effect of coagulant concentration on Cr reduction

Lead (Pb)

Pb is a heavy metal that is toxic or poisonous even in low concentrations. This heavy metal can accumulate in the human body and is bio accumulative. The initial Pb content in the wastewater was 1.93 mg/L which did not meet the quality standard of Pb, i.e. 0.1 mg/L. Figure 6 shows the most optimum Pb reduction of 78.76% reaches when using 80 ppm of PAC coagulant. It can be seen from the graph, the more doses of coagulant used, the higher the Pb reduction. In the coagulation process, there is a destabilization of colloids and particles in the wastewater caused by the addition of coagulant. This destabilization process occurs, because when coagulant is added to the waste, it decomposes into positive and negative ions and can then neutralize colloidal particles as well as bind these particles to form flocs. The floc that is formed over time will become larger and settle based on the force of gravity (Sutapa, 2014).

Zinc (Zn)

On the basis of the initial testing of the wastewater, the Zn value of 95.4 mg/L was obtained, which is beyond of quality standard of Zn, i.e. 5 mg/L. The highest reduction of 69.21% was found by using 60 ppm of ACH coagulant, as demonstrated in Figure 7.

Iron (Fe)

Iron is a metal derived from iron ore (mine) which is widely used for everyday human life. The

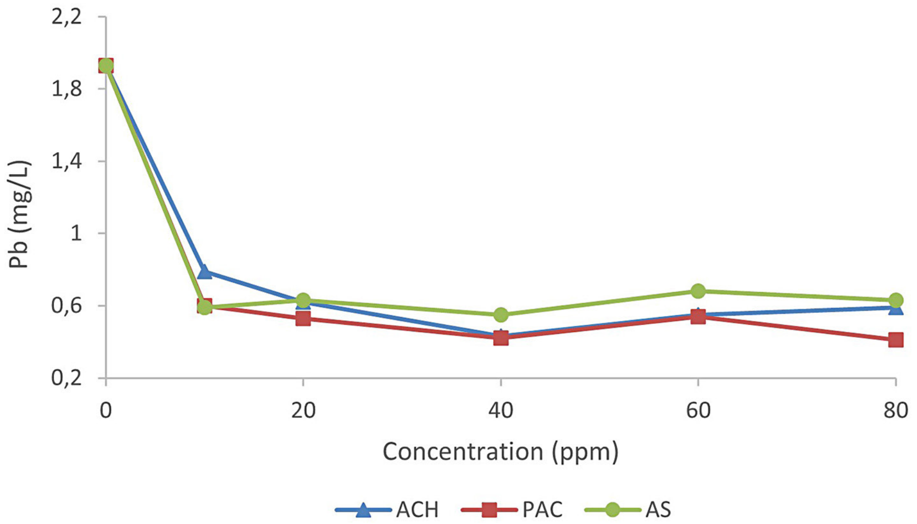

Figure 6. Effect of coagulant concentration on Pb reduction

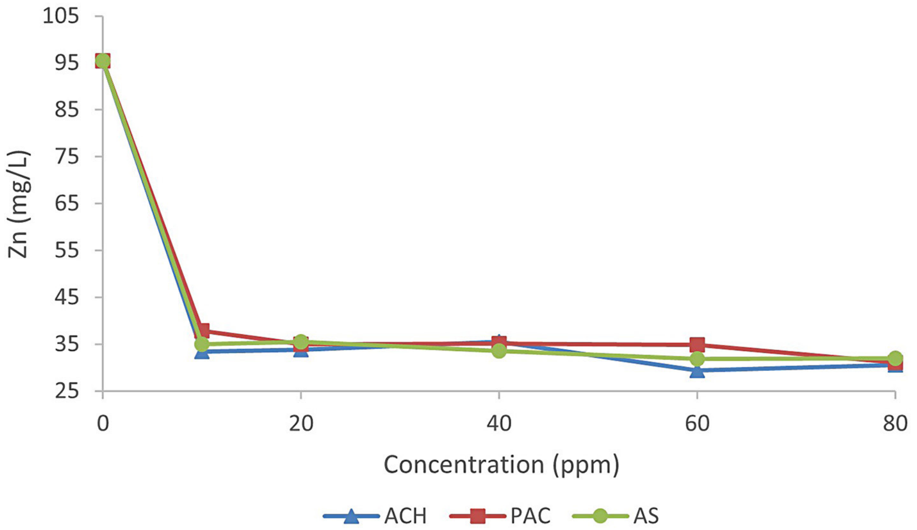

Figure 7. Effect of coagulant concentration on Zn reduction

wastewater contained dissolved iron in the form of ferrous (Fe^{2+}). Iron in the ferrous form is easily oxidized to iron in the ferreous form (Fe^{3+}) in the presence of oxygen in the air (Febrina and Astrid, 2014). High levels of iron in the human body will cause diseases such as poisoning, cancer, liver and homochromatic. On the basis of the initial testing of laboratory wastewater, the Zn value was found to be 60.5 mg/L which exceed the quality standard of Zn, 5 mg/L. Figure 8 above shows the most optimum Fe reduction of 44.55% was obtained when using 10 ppm of ACH. The higher the dose of PAC and AS coagulant added, the higher the percentage of metal reduction. This increase was because the more coagulants were added, the more flocs formed (Nurhayati et al., 2018). Therefore, more metal can be separated from the wastewater. However, at some point, the percentage of metal separation will decrease again.

The level of effectiveness in reducing metal concentration was different between ACH, PAC, and AS coagulants. This is thought to be related to the content and composition of organic compounds contained in the coagulant. The success of coagulation by coagulant material is determined by the content of tightly tension cationic protein.

Coagulation and adsorption

The adsorption process uses two types of adsorbents, namely activated carbon and zeolite for the treatment of laboratory wastewater. The adsorption process is a process that occurs when fluid or liquid binds to a solid substance or liquid, so that it forms a thin layer. The adsorption process was carried out with the use of an activated carbon adsorbent and zeolite for a period of 1 hour and 2 hours of 200 rpm stirring.

The adsorption process was carried out in a batch system using an overhead stirrer device that was used to stir the adsorbent at a predetermined interaction time and stirring speed. The adsorption process was carried out at a room temperature of 28°C; the choice of this room temperature was because the adsorption at the higher temperature caused the less heavy metal ions to be absorbed by the adsorbent. This happens because the higher the temperature in the adsorption process, the faster the movement of the ions, so that the number of heavy metal ions absorbed by the adsorbent will decrease (Faisal, 2015).

The effectiveness of adsorbents can be represented by the average removal percentage of BOD, COD, TSS, Zn, Cu, Fe, Cr and Pb being the given treatment, as reported in Figure 9. The application of activated carbon for 1 hour and 2 hours resulted in adsorption percentage of 50.22% and 23.49%, respectively. Meanwhile, the adsorption percentage of 29.62 and 3.37% were reached, when the zeolite adsorbent was applied for 1 hour and 2 hours of mixing time, respectively.

Figure 9 illustrates that activated carbon in 1 hour mixing time was more effective in reducing the BOD, COD, TSS, Zn, Cu, Fe, Cr and Pb levels, compared to zeolite. This significant decrease occurs as activated carbon has an open pore

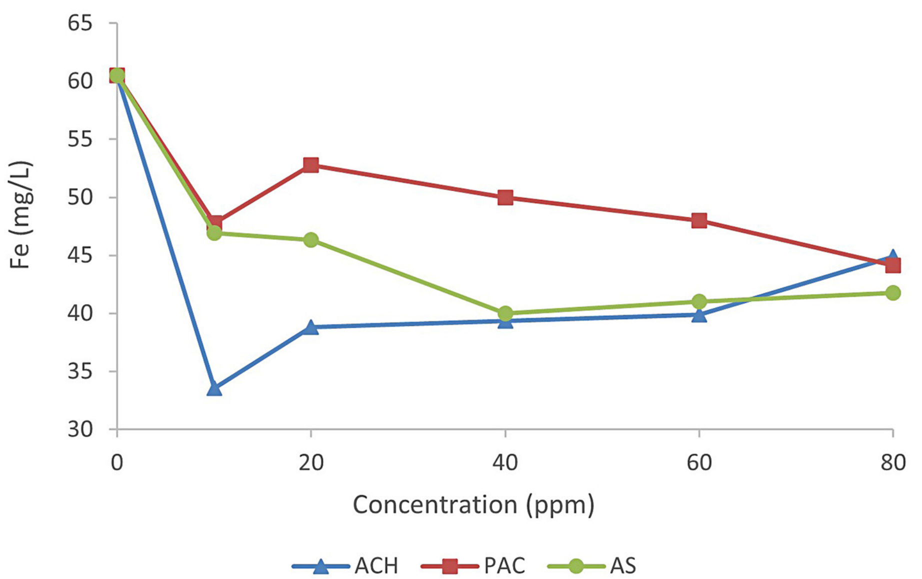

Figure 8. Effect of coagulant concentration on Fe reduction

structure and surface area that allows activated carbon to adsorb molecules in large numbers. This is also because the activated carbon used is finer than zeolite, where the finer the granules used as filtration media, the better the quality of the wastewater that will be produced.

After the coagulation and adsorption processes, only TSS and BOD parameters met the quality standard. The values of COD, Zn, Fe, Cr, Cu, and pH parameter were decreased, but did not meet the quality standard for disposal to the environment. Therefore, the treatment is continued by the Fenton's reagent process to fulfill the environmental quality standard.

Fenton reagent process

In the Fenton reagent process, the pH is adjusted to 3 with the addition of NaOH, because the optimum pH conditions for Fenton's reagents to degrade compounds contained in the waste occur at pH 3. At pH 3, the formation of hydroxyl radicals is maximum so that it can degrade most compounds (Agustina & Amir, 2012). At a pH below 3, the formation of free radicals is less, because the pH is too acidic. Thus, it is possible that iron ions are less optimal in catalyzing the formation of hydroxyl radicals. In turn, at a pH above 3, the decrease in COD is lower because iron ions precipitate so that some hydrogen peroxide (H_2O_2) is unstable and decomposes into oxygen and water, then its oxidizing ability is reduced. The contact time used between laboratory wastewater and Fenton reagent is 60 minutes (Sari, 2019).

The removal percentage of COD, BOD, Zn, Cu, Fe, total Cr, and Pb using Fenton's reagent molar ratio of 1:100 to 1:400 can be found in Figure 10. Based on the figure, the optimum molar ratio of 1:300 was observed in this study. The higher the molar ratio of Fenton's reagent use, the

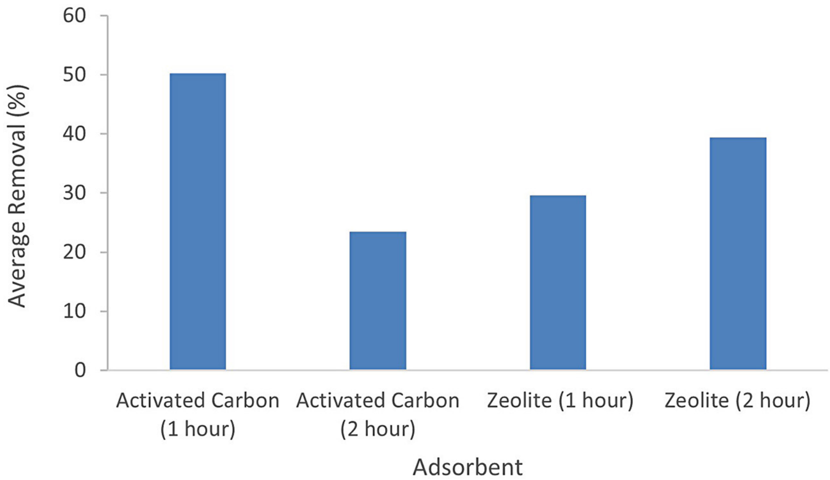

Figure 9. Effect of adsorbent type on average removal percentage

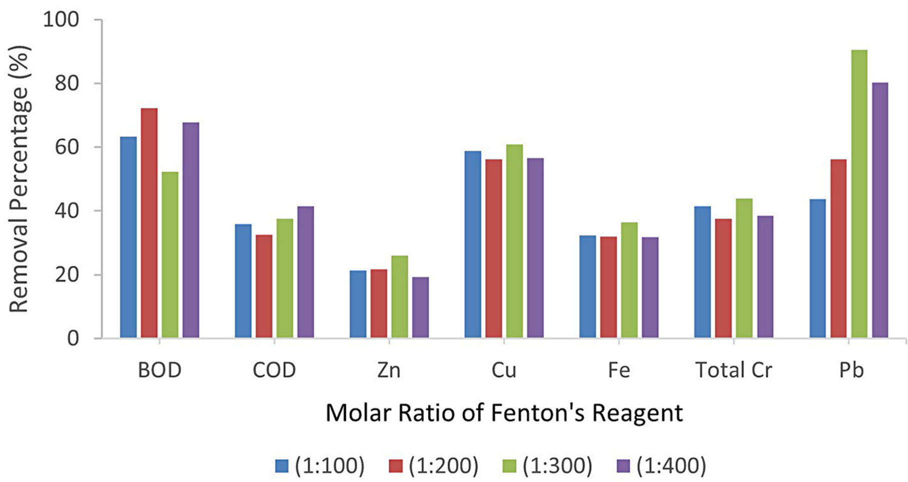

Figure 10. Effect of Fenton's reagent molar ratio on removal percentage of parameters

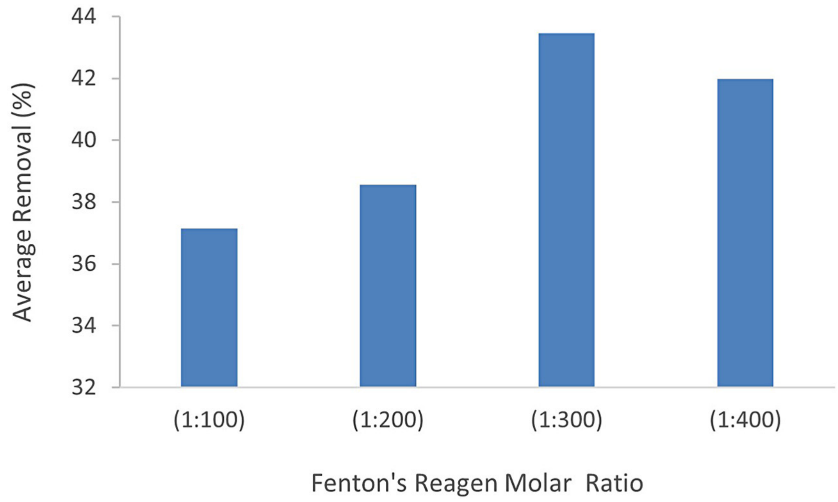

Figure 11. Effect of Fenton's reagent molar ratio on average removal

greater the removal percentage of COD, BOD, and heavy metals is. Because increasing its concentration will increase the concentration of OH radicals, the ability of Fenton's reagent to degrade contaminants in the samples will increase as well.

In Fenton's reagent process, meanwhile, the highest average removal of pollutants parameter of 43.45% was achieved by applying the Fenton's reagent molar ration of 1:300, as shown in Figure 11. However, when using this dose, some parameters were greatly decreased but still did not meet the environmental quality standards such as Zn, Fe, and total of Cr metals. Therefore, it is highly recommended that the treatment be continued with a further adsorption process, so that the metal content can be reduced until it meets the quality standard.

CONCLUSIONS

The type and the concentration of coagulant, the type of adsorbent, and the molar ratio of Fenton's reagent affect the removal of BOD, COD, TSS, and heavy metals parameter. In the coagulation process, the highest average removal of the parameter of 58.21% was attained when utilizing 80 ppm of AS coagulant. In adsorption process, the highest average removal of 50.22% was found by applying activated carbon within 1 hour of mixing time. Moreover, in the Fenton's reagent process, the highest average removal of 43.45% was obtained by using Fenton's reagent molar ratio of 1:300. The average removal pollutant of 90.81% was achieved in the treatment of laboratory wastewater using combined Fenton's reagent and coagulation-adsorption pretreatments. The BOD, COD, TSS, Cu, and Pb contents fulfilled the environmental quality standard. However, even though the metal content of Zn, Fe, and total of Cr are greatly reduced, but they have not met the regulated standard.

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