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Testing of a Laboratory Wastewater Treatment Prototype Using Coagulation, Adsorption, and Photo-Fenton Processes

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ABSTRACT

Research activities carried out in the laboratory produce a complex wastewater which can damage the environmental system if it is directly disposed of without treatment. A laboratory wastewater treatment prototype was designed and constructed to process the wastewater based on the previous research. The prototype is needed in the laboratory to treat the wastewater before discharged into the environment, so it can meet the wastewater quality standards. The wastewater treatment aimed to reduce the pollutant level contained in laboratory wastewater. The objective of this study is to test the efficacy of the prototype. This test was conducted using a combination process of coagulation, adsorption, and photo-Fenton methods. The pollutant parameters were descripted in the form of pH, TSS, COD, BOD, heavy metals of Cadmium, Zinc, Copper, Chromium total, Lead, and Iron. The test of wastewater treatment prototype was carried out using the optimum conditions obtained in previous studies. The best results found from previous studies were attained at laboratory scale by means of ordinary glassware. The results of the initial analysis of laboratory wastewater sample showed that the laboratory wastewater did not meet the wastewater quality standards stipulated in the Minister of Environment regulations of the Republic of Indonesia No.5 of 2014 on Wastewater Quality Standards. In this study, the laboratory wastewater was treated by applied the pre-treatment method of coagulation and adsorption, using alum and activated carbon, separately. By using the wastewater treatment prototype, the removal percentage of COD, BOD, and TSS of 31.47%, 39.90%, and 90.24%, was reached, respectively. The heavy metals content was also reduced, with the removal percentage of Cadmium of 51.30%, Zinc of 33.51%, Copper of 38.43%, Chromium total of 32.61%, Lead of 61.64%, and Iron of 45.83%, were obtained.

Keywords: wastewater, adsorption, coagulation, photo-fenton, wastewater treatment prototype.

INTRODUCTION

Wastewater pollution is a pollution that is very dangerous for living things, especially for aquatic biota when the wastewater is exposed to water. Pollution can damage the aquatic ecosystems that affect human life. Laboratory wastewater is the result of chemical residues used during 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. The laboratory waste can come from various sources such as expired chemicals, broken or damaged consumables, sample residues, residual chemical reaction reagents, and even the used water for washing equipment (Anggraini et al., 2022).

Wastewater generated from research activities can contains heavy metals that need to be handled before being discharged into the environment and prevent environmental pollution. The nature of metals that are difficult to degrade makes heavy metals potentially toxic. Laboratory wastewater besides containing the heavy metals also contains Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), Total Suspended Solid (TSS), Total Dissolved Solid (TDS), and an erratic pH, so it must be treated using coagulation or adsorption methods, or other methods, usually a combined method is also needed in its treatment. Coagulation is known as one of the most mature and effective processes, which can remove most colloids in oily wastewater by forming flocs. In general, the coagulation/flocculation mechanism can be categorized into four important parts, namely simple charge neutralization, charge patching, bridging, and sweeping (Zhao et al., 2020). Adsorption carried out in this study means to reduce the metal levels where the efficiency of reducing the metal levels depends on the type of the metal itself. However, activated carbons (ACs) are often used as adsorbents in water treatment plants to adsorb heavy metals due to their microporous structure, large surface area, and chemical complexity (Ahmad and Azam, 2019). The adsorption process is influenced by the ability of the adsorbent to absorb a solution which is also influenced by the pH of the solution related to the protonation and deprotonation of the surface of the active side of the adsorbent (Nurhayati et al., 2018). The adsorbate bound to this adsorbent is only on the surface of the adsorbent (Hasibuan and Marbun, 2018), so the area of the pores affects the effectiveness of activated carbon in binding the pollutant content in wastewater (Sukatiman and Harjunowibowo, 2014). The adsorption process is carried out with activated carbon adsorbents where these adsorbents are usually very porous, so the process of binding components in waste occurs along these pores The use of activated carbon is based on its abundance, low price, and this adsorbent can potentially reduce contaminants in wastewater (Rahmawati et al., 2018).

In addition to using coagulation and adsorption processes, this research was conducted using the Photo-Fenton method, as one of AOPs method. AOPs is a treatment processes of wastewater at room temperature and pressure which involve the formation of hydroxyl radicals ($^{\bullet}OH$) radicals in an amount sufficient for wastewater purify (Hassaan et al., 2017). The Fenton process is a reaction that uses Fe^{2+} ions as a catalyst held in a homogeneous system. Fenton is one of the most effective advanced methods in the degradation process of many types of pollutants in wastewater. The Fenton process can be carried out at room temperature and atmospheric pressure conditions. Fenton is one of the most effective advanced methods in the degradation process of many types of pollutants in wastewater (Agustina et al., 2016; Agustina et al., 2017). Fenton is related to reactions such as hydrogen peroxide with iron ions. In the Fenton method, hydrogen peroxide is an oxidizer, and iron is a catalyst. Hydroxyl radicals ($^{\bullet}OH$) can be generated from the reaction between iron ions and hydrogen peroxide. The oxidation process will be more effective with additional exposure to UV light which has an additional effect on color removal, which is called the Photo-Fenton process. The reacting UV light produces additional hydroxyl radicals to oxidize toxic organic pollutants in wastewater. Previous studies have proven that the addition of the UV method to the Fenton process has shown better results in the textile industry wastewater treatment (Agustina et. al., 2019). The addition of UV for degradation of synthetic dye waste using Fenton's reagent also proved to be faster and more effective (Agustina et. al., 2016).In the Fenton process, a reaction between Fe^{2+} ions and H_2O_2 produces OH radicals, OH^- and Fe^{3+} (reaction 1). Fe^{3+} can inhibit the formation of hydroxyl radicals because it will inhibit the decomposition of H_2O_2 and form unwanted hydroperoxyl radicals ($HO_2^{\bullet}$) (reaction 2). Photocurrent in the Photo-Fenton process enhances the photo-reduction of Fe^{3+} (reactions 3, 4) because the limited regeneration rate of Fe^{2+} inhibits the Fenton process. Reduction and production of hydroxyl radicals can be achieved simultaneously in the photo-Fenton process (reaction 5) (Liu et al., 2018), as the following reactions:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + {}^{\bullet}OH \quad (1)$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2^{\bullet} + H^+ \quad (2)$$

$$Fe^{2+} + H_2O_2 \rightarrow Fe(OH)^{2+} + {}^{\bullet}OH \quad (3)$$

$$Fe(OH)^{2+} + h\nu \rightarrow Fe^{2+} + {}^{\bullet}OH \quad (4)$$

$$Fe^{3+} + H_2O_2 + h\nu \rightarrow Fe^{2+} + H^+ + {}^{\bullet}OH \quad (5)$$

The UV light used in this study has a wavelength of 370 nm. The difference contained in the Fenton and photo-Fenton processes is in the process of releasing iron deposits and the ability of Hydrogen Peroxide to produce hydroxyl radicals only with the help of UV lamps (Rozas, 2010).

Hydroxyl radicals produced from the Fenton reaction can degrade pollutants in wastewater such as laboratory wastewater. The more $^{\bullet}OH$ free radicals are formed, the more organic pollutants are degraded.

MATERIALS AND METHOD

The laboratory wastewater used come from the Pharmaceutical Analysis Laboratory, Department of Pharmacy, Universitas Sriwijaya. Before testing, the laboratory wastewater was analyzed to determine the pollutant load contained in it. While the chemicals used were activated carbon, hydrogen peroxide, caustic soda, $FeSO_4.7H_2O$, alum, sodium thiosulfate, sulfuric acid, and pharmaceutical laboratory wastewater sample.

The dose used at each treatment stage was the optimum result obtained from previous studies. As reported by Saxena et.al (2020), there were several mixing variables such as turbulence, circulation patterns, detention time, and active zones that can not be taken into account in laboratory jar tests. Whereas in the scale-up to a larger scale, these variables greatly affected the treatment results of the water treatment plant to obtain a similar removal efficiency as in the jar test. Therefore, the dosage was increased by three times and the stirring by 500 rpm.

Coagulation process

Laboratory wastewater was measured as much as 10 L. The laboratory wastewater sample was put into the coagulation tank, then added the alum coagulant as much as 480 ppm/liter. The coagulant dose applied, after multiplied three times of those found from previous study (Arita et al., 2022). Furthermore, the laboratory wastewater that had been added with alum was stirred using an agitator. Stirring was carried out at 500 rpm for 15 minutes. Then, it was continued with slow stirring of 125 rpm for 15 minutes. The wastewater was then allowed to stand for 30 minutes for settling to occured. The treatment results were then filtered and analyzed to see the decrease in pH, COD, BOD, TSS, and heavy metals content of Cd, Zn, Cu, total Cr, Pb, and Fe.

Adsorption process

After coagulation, the process was continued to the adsorption process by adding activated carbon that has been activated with an optimum dose of 10g activated carbon/liter of wastewater (Arita et al., 2022). Stirring was carried out at 500 rpm for 60 minutes. After the adsorption process completed, the sample was filtered and then analyzed for pH, COD, BOD, TSS, and heavy metals content of Cd, Zn, Cu, total Cr, Pb, and Fe..

Photo-fenton process

Laboratory wastewater that has been adsorbed then undergoes a Photo-Fenton process in a UV reactor. However, in the dark processes (Fenton reaction), the reaction time starts when the solution is injected by hydrogen peroxide. In Photo-Fenton process, the time at which the UV lamp was turned on was considered the zero time, or the beginning of the experiment that was taking place simultaneously with injection of hydrogen peroxide and adding the dose of iron ions (Ebrahiem et al., 2017). In this study, Fenton reagent with $FeSO_4$ $7H_2O$:H_2O_2 molar ratio of 1:300 was put into the wastewater sample. It was stirred at 500 rpm while irradiated using UV light at 370 nm. After 120 minutes, 0.5 mL of $Na_2S_2O_3.5H_2O$ was added to stop the reaction. The sample was taken to analyze the pH, COD, BOD, TSS, and heavy metals content of Cd, Zn, Cu, total Cr, Pb, and Fe.

Final adsorption

The second adsorption process were carried out after the photo-Fenton process as further processing due to sludge formed in photo-Fenton (Benatti et al., 2009). In the final adsorption process, the activated carbon was added with the same dosage and stirred as the first adsorption process. After the final adsorption process was complete, the samples were filtered and analyzed for pH, COD, BOD, TSS, and heavy metals content of Cd, Zn, Cu, total Cr, Pb, and Fe.

The overall dimensions of the prototype were 2.6 meters long, 0.5 meters wide, and 1.95 meters high. The prototype was divided into six parts; two of wastewater storage tanks (T-01 and T-06) used 60 L HDPE containers, which are put at the initial and at the end of stages; and four 20 L-acrylic cylinders in which each equipped with an IKA overhead stirrer using a centrifugal agitator mounted on the top of the tank. For the coagulation and adsorption tanks (T-02, T-03, and T-05), acrylic cylinders with dimensions 30 cm high and 23 cm in diameter were used. The bottom of the cylinder is made conical with a height of 10 cm, where at the front and bottom of the cylinder is given a hole with a diameter of 1 inch for the installation of pipes and valves. The same dimension cylinder was used for the UV reactor in photo-Fenton stage (T-04), installed with an aluminum box to prevent UV light from escaping. On the side of the box are vertically mounted three Philips Acnitic 15 Watt UV Lamps that emit the UV-A radiation in the 350–400 nm range. The lamps have a maximum overall length of 45.16 cm and a bulb diameter of 2.8 cm. The prototype set up can be descripted in Figure 1 as below

RESULTS AND DISCUSSION

Effect of coagulation process on pH, COD, BOD, TSS, and heavy metals

The selection of coagulant doses and wastewater pH for effective coagulation is a standard procedure in choosing the optimum pollutant removal conditions (Michel et al., 2019). Therefore, in this study the dose of coagulant was tripled from the previous study conducted on laboratory scale. The pharmaceutical laboratory wastewater sample had an increase in pH after going through the coagulation process. The pH experienced an increase from 2.1 to 2.6. A coagulation process was able to destabilize suspended particles, so the repulsive force between particles contained in wastewater decreased, and flocs with a larger size formed and then settled. The coagulation process reduced COD, BOD, TSS, and heavy metals content in the wastewater. The TSS decreased of 17.07%, from 41 mg/L to 34 mg/L. COD decreased of 12.56%, from 17,134 mg/L to 14,982 mg/L. BOD decreased of 6.91%, from 6,060 mg/L to 5,641 mg/L, and other heavy metals content also decreased after coagulation process.

Effect of initial adsorption process on pH, COD, BOD, TSS, and heavy metals content

The adsorption process was carried out using activated carbon that had previously been activated with NaOH and burned at 500°C for 120 minutes to open and enlarge the pore diameter that had been formed in the carbonization process. The adsorption procedure wass carried out by adding activated carbon to laboratory wastewater which was then stirred using a motor with an agitator at 500 rpm for 60 minutes. In the first adsorption of laboratory wastewater, there was a significant increase in pH after adsorption from 2.6 to 9.2. TSS decreased from 34 to 9 (decreased of 73.52%), COD decreased from 14,982 to 13,963 (decreased of 6.80%), and BOD decreased from 5,641 to 4,852 (decreased

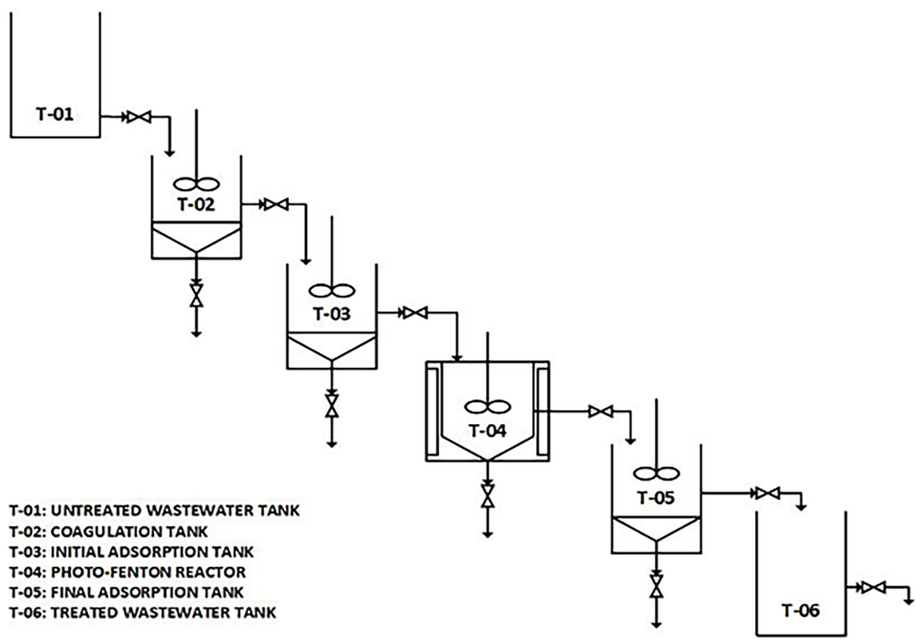

Figure 1. The prototype of laboratory wastewater treatment

of 13.98%). The content of other heavy metals also decreased in accordance with the working principle of the adsorption process where metal contaminant particles were attracted and bound.

Effect of photo-Fenton process on pH, COD, BOD, TSS, and heavy metals content

Fenton is a well-proven oxidative system that oxidizes recalcitrant organic contaminants in effluents by utilizing strong hydroxyl radicals generated in-situ through the reaction of iron with hydrogen peroxide (Diya'uddeen et al., 2015). Photo-Fenton treatment involved radicals from the reaction between hydrogen peroxide and iron ions in the presence of any light irradiation which had a strong oxidizing ability to various organic compounds. The photo-Fenton system based on the photochemistry of iron complexes and the Fenton reaction.

In this research, photo-Fenton reduced COD from 13,963 mg/L to 13,056 mg/L (6.50% decrease), BOD from 4,852 mg/L to 4,044 mg/L (16.65% decrease). Some other heavy metal contents also decreased in accordance with the working principle of UV-assisted photo-Fenton oxidation.

The value of pH is extremely affecting the oxidation potential of free radicals. Moreover, the inorganic carbon concentration and the hydrolytic speciation of iron ions are powerfully affected depending on the pH value. Therefore, the pH impact in the photo-Fenton oxidation process should be strong-considered. The pH value affects the formation of free radicals and therefore, the efficacy of the oxidation process (Hassan et al., 2022; Agustina et al., 2022). A recent literature survey indicated an increased activity in the field of photo-Fenton processes at near neutral pH (Clarizia et al., 2017), which is used in this research. However, some heavy metals also did not decrease because the maximum photo-Fenton reaction was very dependent on dose, pH, and reaction time.

Effect of final adsorption process on pH, COD, BOD, TSS and heavy metals content

The second adsorption was carried out with same method as in the first adsorption. This adsorption was placed as the last treatment to adsorb solid particles and by-products formed after oxidation. In the study reported by Benatti et al. (2009), there was an increase in the Na parameter in both effluent samples treated with Fenton of 67.7 g/L and 92.6 g/L respectively. Furthermore, it was said that although Fenton was an effective technology for chemical wastewater treatment, the sludge produced during the process contained heavy metals that were originally present in the liquid phase and require further separation and treatment. The second adsorption on pharmaceutical laboratory effluent treatment reduced COD from 13,056 mg/L to 11,742 mg/L (removal percent of 10.06%), BOD from 4,044 mg/L to 3,642 mg/L (removal percent of 9.94%), and TSS from 17 mg/L to 4 mg/L (removal percent of 76.47%). Other heavy metals content were also reduced.

Effectiveness of the prototype on COD, BOD, TSS, and heavy metal parameters

COD parameter

The best COD reduction was found in the treatment with coagulation in which there was a decrease of 2,152 mg/L (12.56% removal), while the smallest decrease in photo-Fenton was 907 mg/L (6.50% removal). Overall, the prototype treatment can reduced wastewater COD by 31.47%, as can be seen in Figure 2.

The highest BOD reduction was found in the photo-Fenton treatment in which there was a decrease of 808 mg/L (removal of 16.65%), while the lowest decrease of 6,91% of BOD was found in coagulation treatment. In a whole process, as shown in Figure 3, the series of tools for each treatment reduced the BOD of the laboratory wastewater with a total of removal percentage of 39.90%.

Adsorption process with the activated carbon was very effective in reducing TSS. The first and the final adsorption were the best treatments for reducing TSS, respectively. The removal percentage of 73.53% and 76.47 % were obtained after the first and the final adsorption, respectively. Overall, the TSS removal percentage of 90.24% was achieved by using the prototype, although the TSS had slightly increased in photo-Fenton process, as depicted in Figure 3. Like previous research conducted by Fitriyanto et al., in 2021, the Fenton process reduced BOD and COD content, but increased TS and TSS parameters. This was also seen visually in which the waste after this Fenton treatment turned slightly yellowish. Due

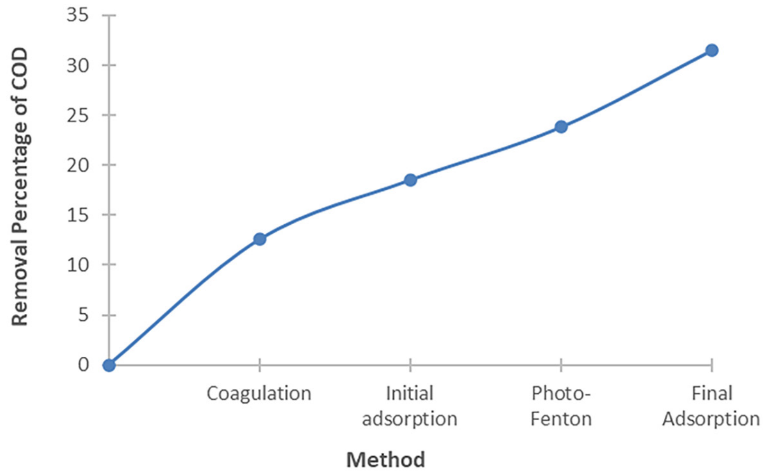

Figure 2. COD reduction after treatment method

to the addition of sulfuric acid to reach an acidic pH before Fenton treatment, it allowed the formation of mineral solids.

Heavy metals content

The heavy metals reduction using different methods of prototype can be shown in Figure 5. The most effective treatment in reducing Cadmium (Cd) was coagulation process, in which 30.57% of removal was found. The prototype can reduce the Cd content with a total removal percentage of 51.30%. The treatment that showed the best results in reducing Copper (Cu) metal content was coagulation in which there was a decrease of 1.09 mg/L or 15.29% of removal. Overall, the device reduced the Cu parameter with removal of 38.43%. The total of Chromium (Cr) parameter was able to be reduced with a total removal of 63.53% using this prototype. The most effective treatment to reduce total Cr in laboratory wastewater was the coagulation process which reduced the total Cr from 1.7 mg/L to 1.02 mg/L (40% of removal). Also, the Iron (Fe) heavy metal decreased the most in coagulation treatment in which there was a decrease of 1.91 mg/L or 28.42% of removal. The percentage removal of Fe metal in laboratory wastewater treatment using this prototype of 45.83% was reached. However, the photo-Fenton treatment was the most effective treatment to reduce the Zinc metal content. Overall, by using the prototype of laboratory wastewater treatment, the removal percentage of 33.51% of Zinc was obtained. One of the heavy metals concentrations that effectively reduced in the wastewater by using this prototype is the Lead (Pb) metal. The percentage removal of total Pb throughout the overall treatment was 61.64%, where most of the reduction occurs in the photo-Fenton process.

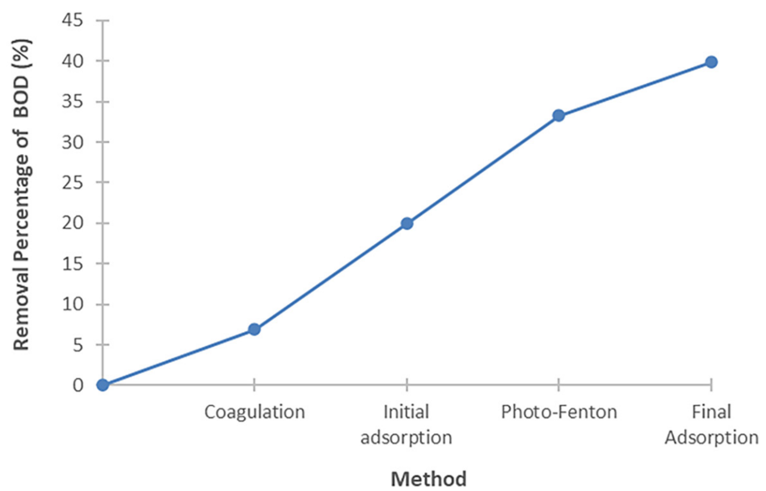

Figure 3. BOD reduction after treatment method

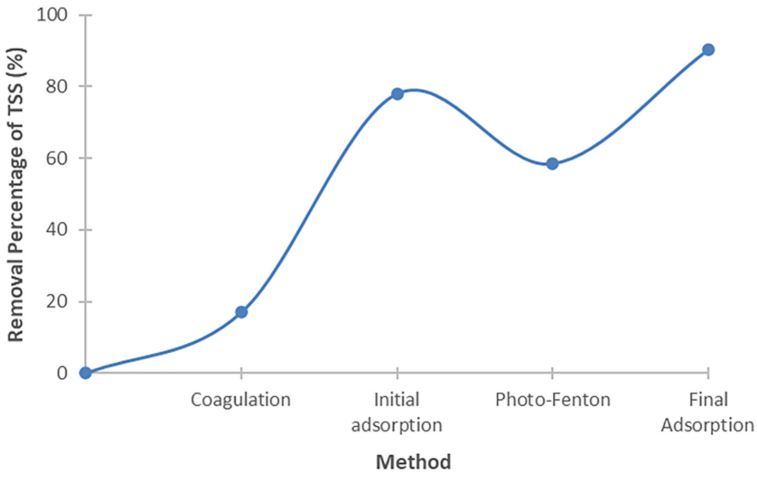

Figure 4. TSS reduction after treatment method

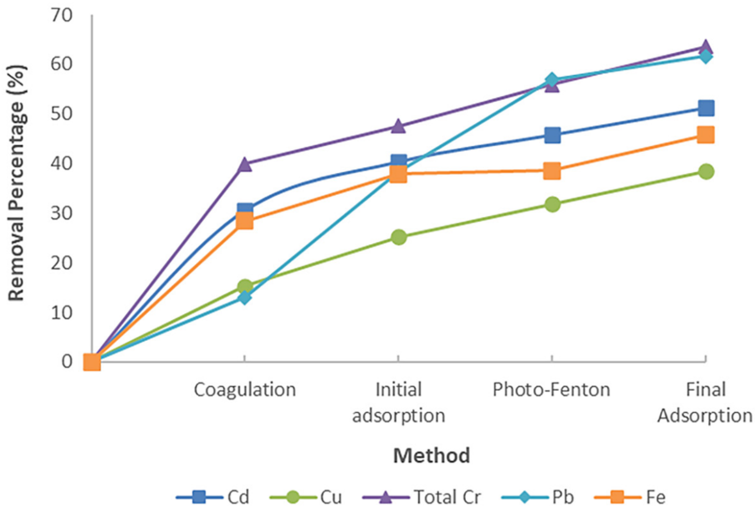

Figure 5. Heavy metals reduction after treatment method

CONCLUSIONS

The combined treatment method had a higher effectiveness when compared to wastewater treatment using only one treatment in which the coagulation process was the most effective treatment to reduce COD, Cd, Cu, Total Cr, and Fe; while the adsorption process was the most effective treatment to reduce TSS; and photo-Fenton was the most effective treatment to reduce BOD, Zn, and Pb. The laboratory wastewater treatment prototype can reduce the pollutant content of Pharmaceutical Analysis Laboratory wastewater sample by a removal percentage COD of 31.47%, BOD of 39.90%, TSS of 90.24%, Cd of 51.30%, Zn of 33.51%, Cu of 38.43%, Cr total of 63.53%, Pb of 61.64%, and Fe of 45.83%. The parameters of TSS, and the heavy metals of Zn, Total Cr, and Fe have met the environmental quality standards according to Minister of Environment regulations.

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