EEET ECOLOGICAL ENGINEERING & ENVIRONMENTAL TECHNOLOGY

Ecological Engineering & Environmental Technology 2024, 25(1), 216–226
https://doi.org/10.12912/27197050/175112
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Received: 2023.11.04
Accepted: 2023.11.28
Published: 2023.12.14

Laboratory Wastewater Treatment by Using Combination Methods of AOPs and Chemical-Physical Pretreatments

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ABSTRACT

Experimental activities carried out in laboratories usually produce complex wastewater. Due to practicum and research activities in educational laboratories, the wastewater generating from these laboratories contains organic and inorganic compounds which are dangerous for the environment if disposed of without prior treatment. Apart from high chemical oxygen demand (COD) and biological oxygen demand (BOD) values, laboratory wastewater also often contains heavy metals such as zinc (Zn), copper (Cu), chromium (Cr), lead (Pb), and iron (Fe) which are included in the hazardous waste category and can pollute the ground water. Therefore, this wastewater must be treated properly. The objective of this study is to reduce the pollutant load contained in laboratory wastewater by using combination methods of advanced oxidation processes (AOPs), and chemical-physical treatment namely coagulation and adsorption processes. The photo-Fenton process was selected as one of AOPs applied in this treatment. The effect of molar ratio variation and irradiation time in the Photo-Fenton process on the pollution load in the form of pH, COD, BOD, TSS, and heavy metals of Zn, Cu, Cr, Pb, and Fe was studied in this research. The results of the analysis of untreated laboratory wastewater samples showed that laboratory wastewater did not meet the wastewater quality standards regulated by the government of Republic of Indonesia. In this study, laboratory wastewater was treated using the pretreatment method of coagulation with alum and adsorption with activated carbon. The best results in this study were obtained in the final adsorption results after treatment with the photo-Fenton method using a molar ratio of 1: 300 for 60 minutes in which several parameters such as pH, Zn, Cu, Pb, and Fe had met environmental quality standards with the value of each parameter of 7; 0.01 mg/L; 2.9 mg/L; 0.03 mg/L; and 3.15 mg/L respectively. Meanwhile, the percentage reduction of COD, BOD, and TSS parameters was 87.49%, 87.02%, and 72.45% respectively.

Keywords: laboratory wastewater, AOPs, coagulation, adsorption, photo-fenton.

INTRODUCTION

Activities contained in the research and educational laboratories produce wastewater that is sometimes not treated properly. The wastewater originated from research activities may contains heavy metals that need to be handled before being discharged into the environment and prevent environmental pollution. Laboratory wastewater besides containing the heavy metals, such as Zn, Cu, Cr, Pb, and Fe, also contains high value of COD, BOD, and total suspended solid (TSS) (Novia et al., 2023). The nature of metals that are difficult to degrade makes heavy metals potentially toxic. The laboratory wastewater sample used in this research comes from one of teaching laboratory in Technical Implementation Unit of Integrated Laboratory. The wastewater sample has a COD value of 34,690 mg/L, BOD value of 12,572, and the metal content of 39,6 mg/L of Cu, 6,1 mg/L of Cr, and 1,4 mg/L of Pb. It shows that not only the COD and BOD value, but also the heavy metal level in the sample is very dangerous, because they exceed the permissible threshold regulated by the Ministry of Environment of Republic of Indonesia.

In this study, to treat the laboratory wastewater, it is necessary to apply the pretreatment to reduce as much as possible the suspended solid and organic pollutant. This is supported using coagulation followed by adsorption method. The coagulation process in this research was carried out to combine small particles into a floc with the help of a coagulant which is then separated through solid-liquid separation (Jiang, 2015). The coagulant that is inserted must be evenly distributed so that all particles contained in the liquid can settle and be separated (Aprilianti and Wahyudi, 2020). 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 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 that the area of the pores affects the effectiveness of activated carbon in binding pollutant content in wastewater (Sukatiman and Harjunowibowo, 2014). Meanwhile, the adsorption process is carried out with activated carbon adsorbents where these adsorbents are usually very porous so that the process of binding components in the waste occurs along these pores (Pungut et al., 2021). 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).

Based on the previous study conducted by Arita et.al in 2022, the average removal pollutant of 90.81% was achieved in the treatment of laboratory wastewater using combined Fenton reagent and coagulation-adsorption pretreatments. The BOD, COD, TSS, Cu, and Pb content has fulfil the environmental quality standard. However, even though the metal content of Zn, Fe, and total of Cr greatly reduce, but they have not met the standard of government regulation. Hence, in this research, in addition to using coagulation and adsorption pretreatment methods, a stronger AOPs were applied, namely photo-Fenton method. Photo-Fenton is one of AOPs where this process produces hydroxy radicals that can degrade wastewater (Atalay and Ersoz, 2016). Modification of Fenton process are called by photo-Fenton. In this process, photo-Fenton can generate hydroxyl radical more than Fenton process (Muthukumar and Babuponnusami, 2013). Hydroxyl radical is one of the pure and reactive chemicals to degrade organic molecules contained in wastewater where this process can be increased in effectiveness by adding catalysts and UV light (Ghime and Ghosh, 2020). Hydroxyl radical is an effective and reactive compound for oxidating pollutant agent in wastewater treatment, which is has a potential oxidation in amount of 2,8V in pH 0 and 1,95 V in pH 14. Hydroxyl radical can break the bond of organic pollutant by radical addition, hydrogen abstraction, electron transfer and radical combination (Deng and Zhao, 2015). The photo-Fenton process is one method that can produce more hydroxyl radicals when compared to the Fenton method. It is hoped that the laboratory wastewater treatment with better results can be achieved by using the photo-Fenton method. This is because in the photo-Fenton method, photo-reduction of iron precipitates into iron (II) ions which then act again as a catalyst to produce more hydroxyl radicals due to the assist of UV light.

The UV light used in this study has a wavelength of 370 nm. The difference contained in the Fenton process and photo Fenton is in the process of degrading iron deposits and the ability of hydrogen peroxide which can produce hydroxyl radicals by assisting of UV lamp. However, the use of sunlight can replace the use of UV lamps to reduce electricity consumption (Agustina et al., 2022). This makes it possible to develop sunlight-based photo-Fenton in countries that have abundant sunlight such as Indonesia. The more hydroxyl produced the more effective the degradation of complex compounds contained in wastewater is. Application of AOPs methods generally can degradation organic compound by oxidation process and remove the inorganic pollutants by the process. AOPs method has a high efficiency and eco-friendly to degradation pollutant which has high toxicity level (Swaminathan et al, 2013).

MATERIALS AND METHODS

Materials

The laboratory wastewater used come from the Organic Chemistry Laboratory at the Integrated Laboratory of Universitas Sriwijaya. The wastewater samples that had not been treated were analysed first to determine the pollutant load contained in them. In this study, the tools used were glass beaker, magnetic stirrer, stopwatch, Erlenmeyer, blender, UV lamp, jar test, fume hood, analytical balance, digital balance, separating funnel, filter paper, furnace, and oven. While the raw materials used were aquadest, alum, activated carbon, $FeSO_4 \cdot 7H_2O$, hydrogen peroxide, NaOH, and $Na_2S_3O_3 \cdot 5H_2O$. The laboratory wastewater was tested for the content of heavy metals pH, COD, BOD, TSS, Zn, Cu, Pb, Cr, Pb, Fe.

Coagulation

Laboratory wastewater was prepared as much as 2 L in a 2L beaker and then added with 320 ppm of alum coagulant (Arita et al., 2022). Furthermore, laboratory wastewater that had been added with alum was stirred using a jar test. Stirring was carried out using the fast stirring and slow stirring methods. Fast stirring was carried out at a rotation speed of 200 rpm for 10 minutes and slow stirring was carried out at a rotation speed of 50 rpm to form flocs. Then, the results of the previous process were precipitated and filtered and then analysed for pH, COD, BOD, TSS, Zn, Cu, Cr, Pb, and Fe parameters.

Adsorption

The first adsorption process was carried out after the coagulation process. The sample after coagulation was added with activated carbon 1:100 and then stirred at 200 rpm for 1 hour. After the adsorption process was complete, the sample was filtered and then analysed for pH, COD, BOD, TSS, Zn, Cu, Cr, Pb, and Fe.

Photo-fenton

The result of adsorption proses were then continued with the photo-Fenton process. In this process, the molar ratio of Fenton reagent $FeSO_4 \cdot 7H_2O:H_2O_2$ was varied from 1:100-1:300. The wastewater sample was put into a reactor equipped with a UV lamp. Time recording began after the lamp was turned on. Sampling was carried out every 15 minutes for 1 hour. When the photo Fenton process was complete, add the sodium thiosulfate to stop the reaction. Wastewater samples were filtered and analysed for pH, COD, BOD, TSS, Zn, Cu, Pb, and Fe.

Final adsorption

The second adsorption process was carried out after the photo Fenton process. The sample after photo Fenton was added with activated carbon 1:100 then stirred at 200 rpm for 1 hour. After the adsorption process was complete, the sample was filtered and then analysed for pH, COD, BOD, TSS, Zn, Cu, Cr, Pb, and Fe.

RESULTS AND DISCUSSION

Effect of coagulation method on pH, COD, BOD, TSS, Zn, Cu, Cr, Pb, and Fe levels

Alum coagulant was able to reduce pH in the processing process due to the contact between the coagulant and negative ions to form flocs. The floc granules then settled as the settling time passed and lowered the pH. Alum coagulant used had the ability to release hydrogen groups which then lowered the pH of the wastewater. After the alum dose addition treatment, the pH of the wastewater decreased to 4.49. This was because alum was able to produce H_2SO_4 which then caused the pH to decrease (Tandiarrang, et al 2016). COD and BOD values decreased when added using alum coagulant. This was due to the decrease in the load of organic content in wastewater which resulted in the COD and BOD levels decrease. The increase in TSS occurred due to lack of settling time and imperfect separation during the process.

The decrease in zinc metal contained in wastewater was 56% while the decrease in copper metal in wastewater was 17.17%. Coagulants in the form of aluminium could be hydrolysed in water into positively charged hydro complexes. The hydro complexes formed then formed hydro metal polymers and formed $Al(OH)_3$ precipitates accompanied by an increase in the concentration of H^+ ions in the solution. This positive charge could then neutralize the negative charge contained in the Cr anion so that it could precipitate along with $Al(OH)_3$ (Witono et al., 2015).

Coagulation treatment of laboratory wastewater could reduce heavy metal levels of Lead (Pb). This was because Al^{3+} ions in alum played a role in binding and reducing heavy metal lead in laboratory wastewater with its positive electrolyte in destabilizing colloidal particles. Iron coagulant in the form of iron behaved similar to aluminium sulphate and forms $Fe(OH)_3$ flocs in the presence of bicarbonate alkalinity. Due to the instability of the coagulation and flocculation process that had been carried out, the iron content contained in wastewater changed its role to an unstable coagulant and it was because of the stirring speed factor that could increase the amount of iron ions. The coagulation treatment was able to reduce metal content because metals contained in laboratory wastewater were particulates that could be removed through the coagulation process.

Effect of adsorption method on pH, COD, BOD, TSS, Zn, Cu, Cr, Pb, and Fe levels

The metal ions formed were then pulled by activated carbon with Van der Waals forces so that what was left was hydroxide ions. The interaction of activated carbon metal ions was that metal ions were exchanged for acidic functional groups on the surface of activated carbon so that H^+ ions were reduced so that the pH increased (Heriyani and Mugisidi, 2016). The decrease in COD levels was 6,160 mg/L or a decrease in COD levels of 20.88% after the adsorption process as shown in Figure 1. This was because adsorption using activated carbon was able to bind organic content directly proportional to the amount of activated carbon adsorbent added. The decrease in BOD occurred because the organic substance was absorbed by the adsorbent, absorption. Adsorption carried out for 1 hour using activated carbon was able to reduce BOD content levels significantly to 7,001 mg/L.

In the adsorption process, activated carbon absorbed pollutant molecules in wastewater. This adsorption could occur due to the force field on the surface of the adsorbent that attracted adsorbate molecules. This condition made particles or molecules of contaminants in wastewater stuck to the surface of activated carbon due to the weak charge difference (Van der Waals force) between the two (attraction between the positive charge of activated carbon and carboxyl groups on negatively charged contaminants). Thus, forming a thin layer of fine particles on the surface of activated carbon so that the TSS value dropped to 472 mg/L from 1,357.5 mg/L or a decrease of 88.25%.The optimum conditions in the adsorption process to remove chromium content were at a low or acidic solution pH. This was because the H^+ ions on the adsorbent surface increase, resulting in a strong electrostatic bond on the positive charge on the adsorbent surface with chromium (Utama, 2016). The adsorption process that occurred on activated carbon was a physical adsorption process in which the absorption of metal ions Zn, Cu, Pb, Fe occurred on the surface of activated carbon. With the Van Der Waals force in the pores of activated carbon, the Zn, Cu, Pb, Fe contaminant particles contained in the waste were attracted and trapped in the pores of activated carbon and made metal ions in the waste reduced.

Effect of photo-Fenton method on the decrease of pH, COD, BOD, Zn, Cu, Pb, and Fe

Acidic conditions in the photo Fenton process were one of the main factors for a good photo-Fenton process. The presence of an acidic atmosphere could help accelerate the time hydroxyl radicals react in water. The results shown in Figure 2 showed that at each molar ratio of 1:100, 1:200 at any time and 1:300 at 15 minutes the pH reached 5. Meanwhile, at 1:300 at 30, 45 and 60 minutes the pH increased to 6. This was due to the formation of more $Fe(OH)_3$ and $Fe(OH)^{2+}$ which

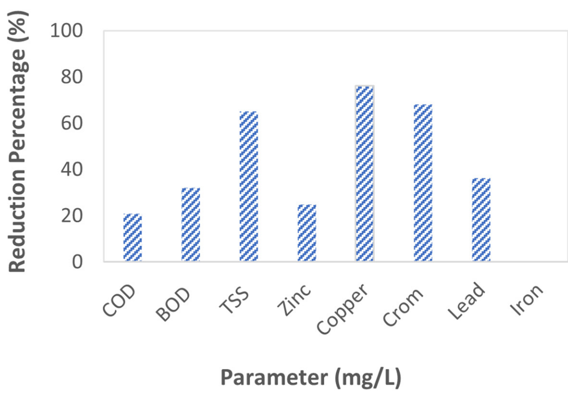

Figure 1. Reduction percentage of pollutant levels in laboratory wastewater

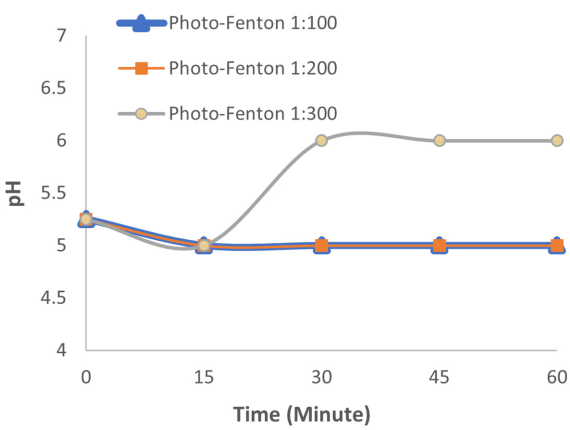

Figure 2. Effect of photo-Fenton method on pH reduction

had an alkaline atmosphere. Besides, more minerals were formed due to the degradation of OH radicals so that the pH increased.

A decrease in COD levels occurred in the photo-Fenton process. This was because the hydroxyl radicals formed during the process could degrade the COD content which made the COD value decreased. The average decreased in COD content from the initial waste at a molar ratio of 1: 100 with a time of 15, 30, 45 and 60 minutes after the photo-Fenton process was 30.06%. The average COD decreased after the process at the same time as before and the molar ratio of 1:200 was 36.49%. For molar ratio 1:300, it was 51.05%. The largest decrease in COD value was in the variation 1: 300 within 15 minutes which was 54.13% or left the COD waste content of 13,527 mg/L as shown in Figure 3. The increase in COD levels in wastewater was due to the decomposition of organic compounds so that the value of COD might be increased. The mineralization of the more dominant organic substance content could be resulted in the decrease of COD level again.

The decrease in BOD levels occurred due to the large number of hydroxyl radicals formed so it was able to oxidize organic matter into simpler elements and could increase processing efficiency. The increase in BOD levels that occurred due to the greater addition of H_2O_2 and the small concentration of $FeSO_4$ caused OH radicals to react with H_2O_2 to produce HO_2 radicals which were less effective for use in waste treatment so that the combination of H_2O_2 and $FeSO_4$ must have been balanced to produce optimum processing (Lesa et al., 2020). The best results of this study were in the variation of the molar ratio of 1: 300 with an average removal of BOD levels of 40.24% (Figure 4) with the most optimal time of 15 minutes obtained removal of 40.25% leaving a content of 4,180 mg/L. The most effective reduction in metal ion concentration was shown by the process involving a combination of Fe^{2+} ions, H_2O_2

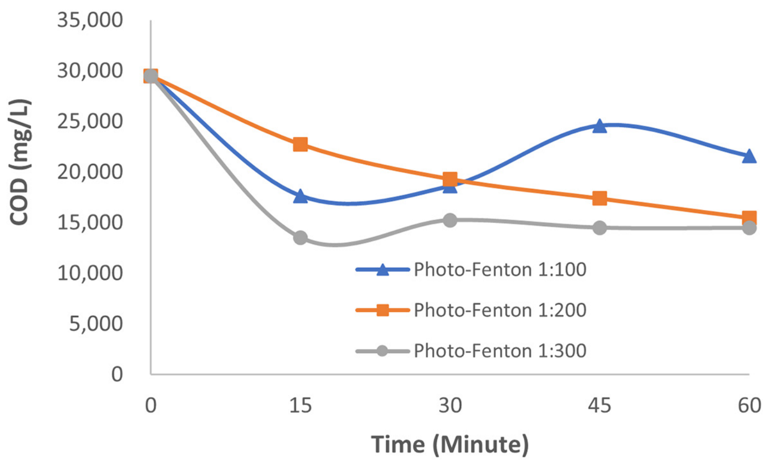

Figure 3. Effect of photo-Fenton method on COD reduction

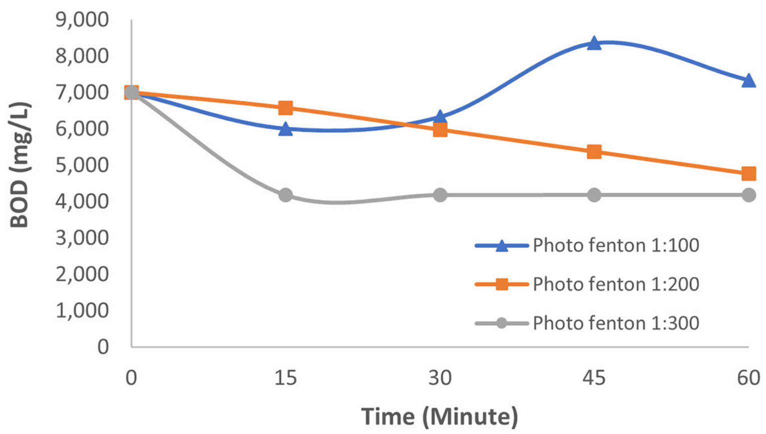

Figure 4. Effect of photo-Fenton method on BOD reduction

and UV light. In this process there was a reaction between Fe^{2+} with H_2O_2 and H_2O_2 with UV light which produced OH radicals and electrons in larger quantities. The decrease in Zn metal levels was shown in the graph above. The average decrease in metal content at each time with a molar ratio of 1:100, 1:200 and 1:300 was 21.66%, 28.33%, and 70.83% respectively as illustrated in Figure 5. The largest decrease in Zn metal content was shown in the ratio of 1:300 with a time of 60 minutes which had a percentage of degradation of wastewater with Zn content of 90% and left Zn metal content in laboratory wastewater of 0.03 mg/L.

The longer the contact between light with H_2O_2 and between Fe (II) ions with H_2O_2, the more OH radicals and electrons were formed more effectively between Cu (II) ions with electrons. This condition could produce photo oxidation and photo reduction reactions were increasingly effective. The increase in Cu (II) levels was due to the presence of Cu solids that were formed. They could block the penetration of light into the solution so that the formation of OH radicals and electrons did not increase (Wahyuni et al., 2019).The most optimum condition in reducing Cu levels was at a molar ratio of 1: 300 with a time of 45 minutes with a copper removal percentage of 41.02% leaving a Cu (II) content of 4.6 mg/L as found in Figure 6. In the research conducted, the removal of copper metal in laboratory wastewater that had been treated using the coagulation, adsorption and photo-Fenton processes still did not meet the wastewater quality standards as described in ministerial regulation of environment No. 5 of 2014 where it was stated that the Cu (II) content was 3 mg/L.

The decrease in Pb (II) ion concentration was the result of oxidation by H_2O_2 which acted as an oxidizer. In this process, OH radicals and electrons generated from photolysis reactions with H_2O_2 by UV light were effective so that the number of OH radicals formed was relatively more. The longer the contact between light with H_2O_2

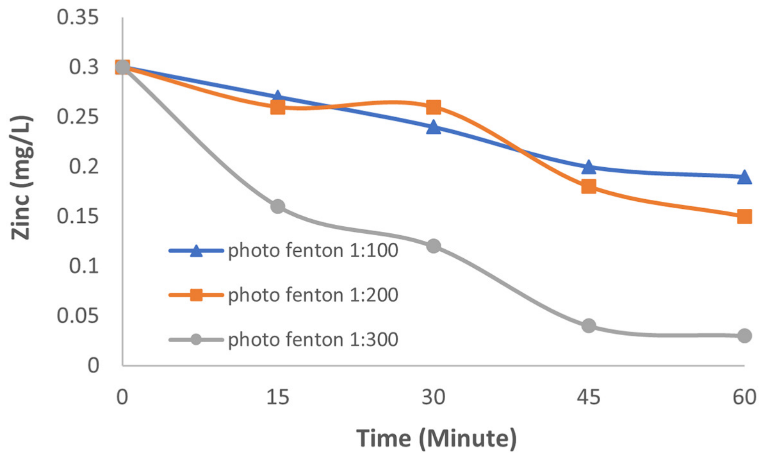

Figure 5. Effect of photo-Fenton method on zinc reduction

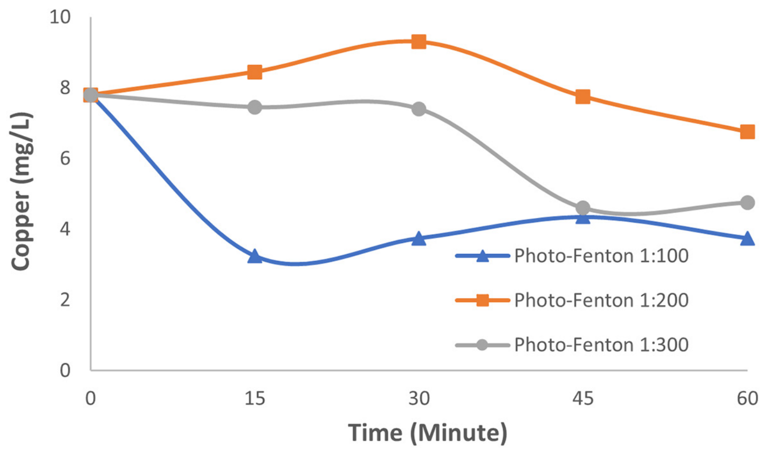

Figure 6. Effect of photo-Fenton method on copper reduction

and between Fe (II) ions with H_2O_2, the more OH radicals were formed, the more effective the reaction of Pb (II) ions with OH radicals. The presence of Cu (II) ions in the Pb (II) solution could increase the photooxidation of Pb (II) ions. This was because the OH radicals and electrons that had been formed during the process could be recombined so that the number of OH radicals decreased, and this could reduce the effectiveness of oxidation reaction of H_2O_2.

In the presence of Cu (II) ions, Cu (II) ions were able to capture electrons so that the recombination between OH radicals with electrons could be prevented and the number of OH radicals did not decrease. This resulted in an increase in the efficiency of the oxidation reaction of Pb (II) ions so that their levels decreased (Wahyuni et al., 2019) along with the large amount of H_2O_2 added to fulfil the oxidation process as a decrease in Pb (II) levels in laboratory wastewater. The optimum condition for the decrease of Pb metal of 95.71% was found when using a molar ratio of 1: 300 within 60 minutes resulting in Pb concentration of 0.03 mg/L as shown in Figure 7.

The process of UV irradiation in the process involving Fenton reagent accelerated the regeneration of Fe^{2+} or commonly known as photo reduction. The photo reduction process occurred when Fe^{3+} absorbed UV light which reduced it to Fe^{2+} again, and then produced more hydroxyl radicals in other word it could more quickly degrade organic waste. Fe deposition decreased due to the help of UV light or photo reduction where the Fe^{3+} formed reacts back to form $Fe(OH)^{2+}$. The decrease in Fe levels in the photo-Fenton process could be seen in Figure 8, which showed a decrease in each Fe contamination with Fenton reagent.

Effect of the second adsorption on pH, COD, BOD, TSS, Zn, Cu, Cr, Pb, and Fe content

Activated carbon used in the adsorption process had the ability to degrade metal elements into metal ions and hydroxide ions. Metal ions were then bound to the surface of activated carbon which had H^+ ions so that H^+ ions were bound to metal ions and H^+ ions were reduced. This caused the pH of the waste to increase. The effect of adsorption on pH could be seen in Figure 9.

The optimal condition was shown by using a molar ratio of 1:300 for 60 minutes where the COD degradation reached 87.49% (Figure 10). Adsorption was known to be able to degrade COD significantly at a Fenton photo molar ratio of 1:300 for 60 minutes. The percentage of COD removal at a molar ratio of 1:300 for 60 minutes in the previous process had been able to degrade the complex organic content of the waste into minerals so that the pollutant load in the wastewater was reduced. In addition, the use of activated carbon size used in second adsorption was larger (granular) when compared to the first adsorption (powder) so that it was possible that the number of pores on the surface of activated carbon was more and could reduce COD levels more significantly than using fine activated carbon because it had fewer surface pores, so that activated carbon was saturated faster.

The decrease in BOD from the adsorption method occurred due to the organic content absorbed by the activated carbon adsorbent. After photo-Fenton processing, it was known that the best BOD reduction value was at a molar ratio of 1:300. However, over time, the waste ratio of 1:300 experienced a significant decrease in BOD. This was because the BOD value previously

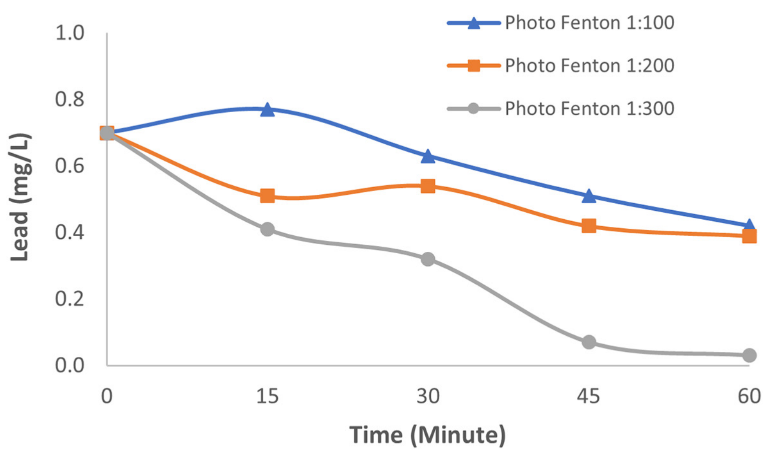

Figure 7. Effect of photo-Fenton method on lead reduction

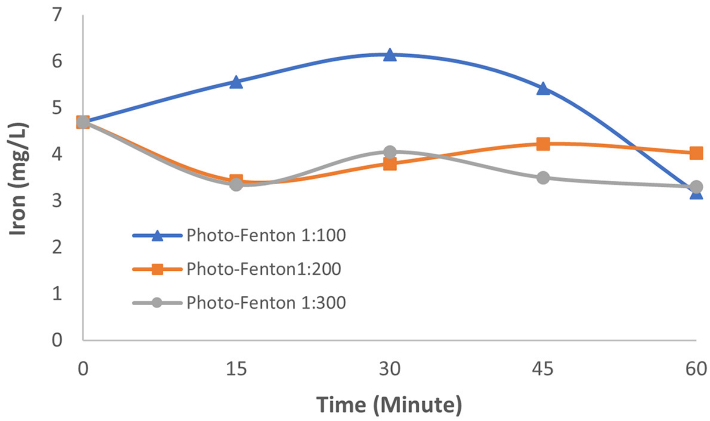

Figure 8. Effect of photo-Fenton method on iron reduction

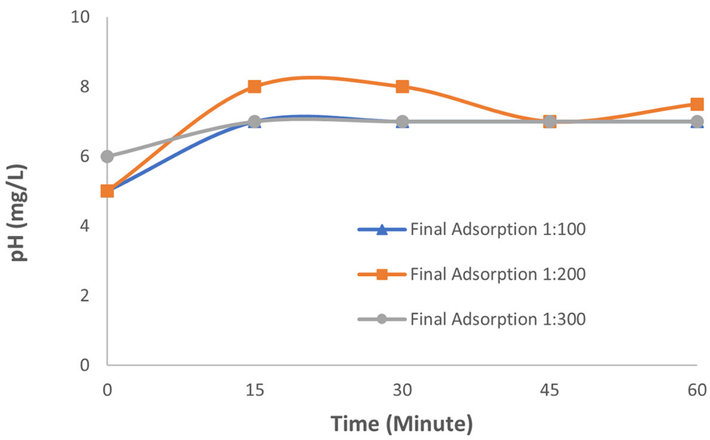

Figure 9. Effect of the second adsorption on pH

treated using photo-Fenton had produced a large enough decrease so that the amount of initial BOD before the final adsorption was less. The percentage of BOD removal in laboratory waste showed that the 1:300 ratio for 60 minutes had a higher percentage of removal compared to other variations. This was because the amount of BOD at 1:300 60 minutes was less than the others and the amount of activated carbon was fixed. The largest percentage was at 87.02% as shown in Figure 11.

The decrease in TSS occurred due to the force of attraction of molecules which made molecules that were adsorbates bound to activated carbon. Activated carbon as an adsorbent had an open pore structure and had a surface area capable of adsorbing several impurity particles in the solution. The precipitate contained in the process after photo-Fenton was more and more due to the help of OH radicals, the greater the precipitate contained in laboratory wastewater after treatment, the greater the TSS value in the wastewater was. The highest percentage of removal was found in the 1:300 variations for 45 minutes and 30 minutes where the percentage of removal was 90.87% and 68.08%, respectively (Figure 12). Metal content was one of the particulates that was difficult to remove using only one method. For this reason, optimization of the process as well as the molar ratio was necessary to produce the optimal results. Combining various methods of removing waste test parameters was expected to produce the best results so that the waste could meet the wastewater quality standards.

The decrease in metal content in wastewater by adsorption method occurred due to the Van der Waals force where there was an attractive force between the adsorbate and adsorbent. However, it could not be denied that the decrease that occurred did not always occur perfectly. This was due to the saturation of the adsorbent used. When the adsorbent was saturated, the adsorbent was no longer able to work optimally to bind the adsorbate on the surface. The optimum conditions in the adsorption process to remove chromium

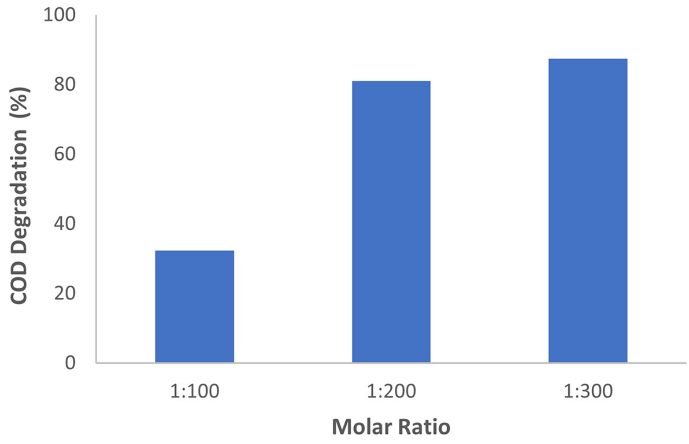

Figure 10. Effect of the second adsorption on COD degradation

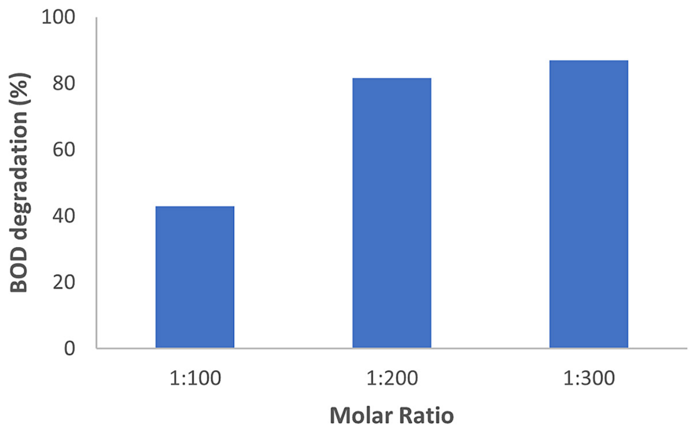

Figure 11. Effect of the second adsorption on the BOD degradation

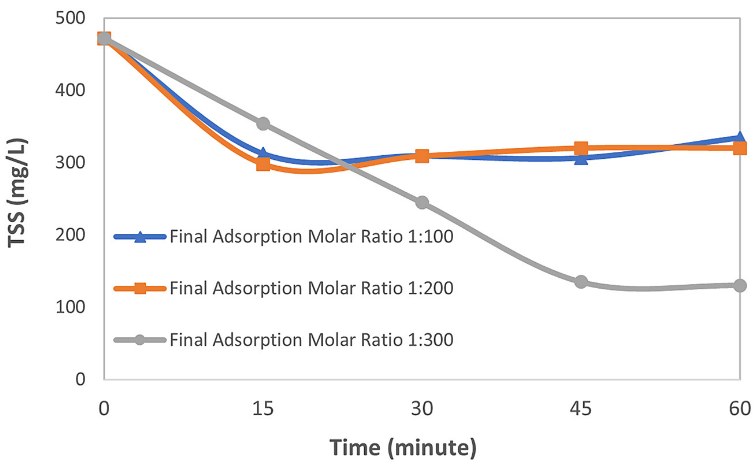

Figure 12. Effect of second adsorption on TSS reduction

content were at a low or acidic solution pH. This was because the H^+ ions on the adsorbent surface increase, resulting in a strong electrostatic bond on the positive charge on the adsorbent surface with chromium so that the chromium content decreased. The instability of the decrease in chromium content was due to its valence which changed and was not constant, making it difficult to analysed precisely. The zinc, lead, and iron content that had been treated using several

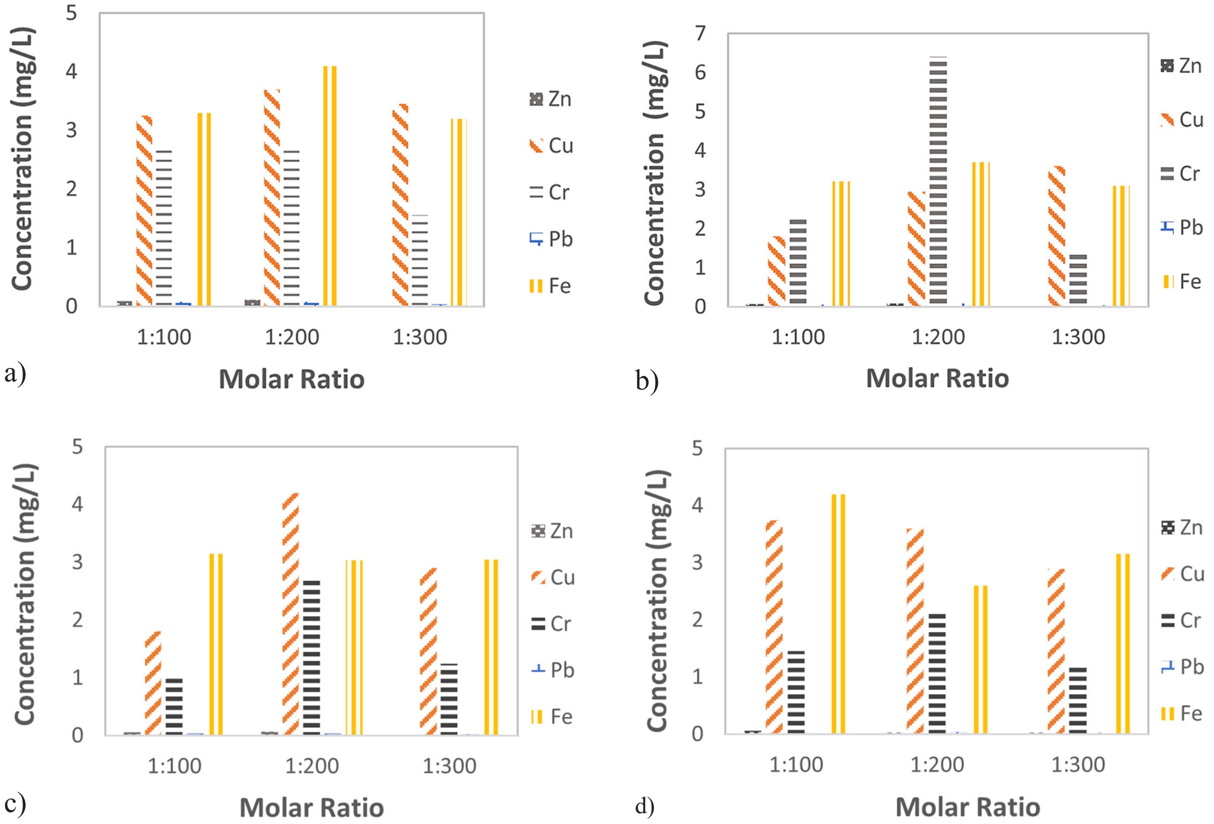

Figure 13. Reduction of Zn, Cu, Cr, Pb, and Fe metal content by second adsorption at (a) 15; (b) 30; (c) 45; and (d) 60 minutes

treatments above made the entire zinc, lead, and iron content with all variations of molar ratios and time meet the wastewater quality standards mentioned in the Minister of Environment Regulation No. 5 of 2014, which was 10 mg/L, 1 mg/L, 10 mg/L respectively. For copper content, the quality standard was at a value of 3 mg/L so the variations that met the wastewater quality standards were at the molar ratio of 1:100 within 30 and 45 minutes, 1:200 within 30 minutes, and 1:300 within 45 minutes. However, the chromium metal content still did not meet the quality standards. The remaining metal levels still contained in the wastewater after the treatment could be seen in Figure 13 respectively, after 15 minutes (a), 30 minutes (b), 45 minutes (c), and 60 minutes (d).

CONCLUSION

The molar ratio of Fenton reagent affected the decrease of COD, BOD, Zn, Cu, Pb, and Fe. The best molar ratio was obtained at a molar ratio of 1:300. The irradiation time affected the decrease in COD, BOD, Zn, Cu, Pb, and Fe levels. The best irradiation time to treat laboratory waste was obtained at 60 minutes. The combination method of coagulation, adsorption, and photo-Fenton was able to reduce Fe levels to 3.31 mg/L, which meant it meet the wastewater quality standards. The photo-Fenton method could be carried out with a higher molar ratio to further degrade COD and BOD content.

Acknowledgments

The author would like to thank the Integrated Laboratory of Universitas Sriwijaya. The publication of this article was funded by DIPA of Public Service Agency of Universitas Sriwijaya 2023. SP DIPA-023.17.2.677515/2023, on November 30, 2022. In accordance with Rector's Decree Number: 0187/UN9.3.1/SK/2023, on April 18, 2023.

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