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Reduction of Heavy Metal (Pb) ions in industrial waste using activated carbon based on used catalysts

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Abstract
Used catalyst-based activated carbon is activated carbon made from used catalysts from the crude oil cracking process. The catalyst used is a type of crystalline Zeolite with a regular structure, which contains elements of Silica Oxide, Alumina Oxide, and Calcium Oxide. The use of organic activated carbon to treat waste has been widely used, the use of used catalyst waste as activated carbon is very potential because the catalyst used for oil company cracking is no longer used. Tofu industry is one of Indonesia's industries dominated by small-scale businesses with limited capital, so most tofu industries do not have waste treatment units, where liquid waste is directly discharged into sewers or water bodies without treatment first. Tofu industrial waste contains pollutants both organic and inorganic. Initial analysis of tofu industry liquid waste containing Pb metal. This study aims to determine the effect of mass variation and stirring time of used catalyst-based activated carbon adsorbent on reducing Heavy Metal Ions (Pb) in tofu industry liquid waste. The adsorption process is carried out with mass variations of activated carbon of 5, 10, 15, 20, and 25 grams. Stirring time variations are 30, 60, 90, 120, and 150 minutes. From the results of the study, it is known that the best condition for reducing Pb content was obtained at a stirring time of 60 minutes and a large catalyst-based activated carbon mass of 66.33%

***Keywords: Activated carbon, cracking catalyst, Adsorbent, Tofu Liquid Waste, Pb metal*.**

Introduction

Tofu industries are widely available in Indonesia and most of them are integrated with residential areas, so problems arise with local residents. The tofu industry produces liquid waste that can result in environmental pollution. Tofu industry activities in Indonesia are dominated by small-scale businesses with limited capital, so most tofu industries do not have sewage treatment units, where liquid waste is directly discharged into sewers or water bodies without treatment first. In liquid waste, know the organic content in the form of (BOD, COD, TOC) dissolved oxygen (DO), oil or fat, total nitrogen, and others. While inorganic chemistry includes: pH, Pb, Ca, Fe, Cu, Na, sulfur, and others. This will cause oxygen levels in the water to decrease sharply. Tofu liquid waste also contains suspended substances, resulting in dirty and turbid water [1]

Various methods that have been widely used to process tofu liquid waste and remove harmful substances contained in tofu liquid waste so as not to pollute the environment. These various methods include: ion exchange methods, reverse osmosis, membrane-based filtration for precipitation in complex form, electrocoagulation, precipitation and adsorption [2]. Generally, adsorption is a simpler method, has high effectiveness, is environmentally friendly and easy to operate compared to several other methods. Various types of adsorbents for physico-chemical adsorption of heavy metal ions have been widely developed such as: clay, zeolite, inorganic polymers, and activated carbon [3]. The adsorption method has attracted the most interest in water treatment due to its simple design, flexibility of operation, low cost and simplicity [4]. Adsorption is a surface phenomenon or separation process in which certain substances are removed from the fluid phase known as gases or liquids, and usually includes gaseous, liquid, and solid molecules in which atoms or ions are attached to the surface in the dissolved phase[5]. The reverse reaction of the adsorption process is known as the desorption process[6].

In the tofu liquid waste treatment research, the author will carry out an adsorption process to remove heavy metal ions using catalyst waste used for the petroleum cracking process. This catalyst has the main components silica and alumina oxide, but also contains sodium, calcium, magnesium and a little Lanthanum. According to the decree of the State Minister of Environment KEP-51 / MENLH / 10/2014 concerning Liquid Waste Quality Standards for Industrial Activities, the level of lead content parameters (Pb^{2+}) allowed is 0.1-1 mg / L. If more than the predetermined threshold limit can be dangerous if wasted into the aquatic environment. This is because lead can accumulate so that the longer the tofu liquid waste containing lead is discarded, the more lead content will be in the waters. Because as we know, there are still many people who depend on the aquatic environment, especially people who live around the waters or suangi. With this content limit, it is necessary to process tofu liquid waste so as not to exceed the quality standards that have been set and in the future the liquid waste produced can be purified first so as to reduce harmful impacts on the environment.

Materials And Methods

Material

The material used in this study is a used catalyst obtained from the oil and gas processing industry as a material for making activated carbon. The activated carbon that has been obtained was carried out a pretreatment process before being made into activated carbon, the used catalyst is cleaned from dirt carried away when the used catalyst is taken. After cleaning, the used catalyst is calcined in the furnace at a temperature of 700 C for 1 hour. The calcined catalyst is cooled in a desiccator. The used catalyst that will be used as activated carbon is 100 mess in size. Industrial liquid waste is obtained from one of the tofu industries in the plaju area of Palembang city. This liquid waste is filtered first using a 0.1 μm filter so that the solids carried in the waste can be separated before the wastewater is treated using activated carbon.

Method

1. Determination of Optimum Mass

Maceration

Prepared respectively as much as 5, 10, 15, 20 and 25 gr of catalyst waste plus 100 ml of tofu industry wastewater. The mixture is precipitated with a constant contact time of 60 minutes. Then separated between the filtrate and the adsorbent, the filtrate is measured Pb concentration value.

Stirring

Prepared respectively as much as 5, 10, 15, 20 and 25 gr of catalyst waste plus 100 ml of industrial wastewater (tofu industry). The mixture is stirred at a constant speed using a magnetic stirrer with a standard contact time of 60 minutes. Then separated between the filtrate and the adsorbent, the filtrate is measured Pb concentration value.

2. Determination of Optimum Time

Maceration

Mixing with the optimum mass ratio obtained from the previous procedure was carried out and then added 100 ml of tofu industrial wastewater. The mixture is precipitated with time variations of 30, 60, 90, 120 and 150 minutes. Then separated between the filtrate and the adsorbent, the filtrate is measured Pb concentration value.

Stirring

Mixing with the optimum mass ratio obtained from the previous procedure was carried out and then added 100 ml of tofu industrial wastewater. The mixture is stirred at a constant speed using a magnetic stirrer with time variations of 30, 60, 90, 120, and 150 minutes. Then separated between the filtrate and the adsorbent, the filtrate is measured the value of the concentration value of Pb.

Characterization of Activated Carbon

Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is a microscope-like instrument that uses high-energy electrons instead of light to produce various signals on the surface of solid specimens. The working principle of SEM

is that when electrons from the cathode are fired towards the sample, there will be an interaction between the electrons and the sample which results in electrons losing some energy.

Energy Dispersive X-Ray (EDX/EDS)

The specific energy of X-rays emitted by each atom in a compound can be detected by Energy Dispersive X-Ray Spectroscopy (EDX). EDX is an analytical technique used to analyze elements or characterize the chemical content of a sample. EDX analyzes X-ray emission by elements in particles. To find out the elements in the sample, by firing X-rays at the position that you want to know the composition. This SEM-EDX / EDS analysis technique can be used quantitatively analysis of the percentage of each elemental element contained in the sample tested.

Fourier Transform Infrared (FTIR)

Fourier Transform Infrared (FTIR) is an instrument used to identify compounds based on the vibrations of atoms in molecules. An infrared spectrum is usually produced by passing infrared light through a sample so that it can determine the fraction of radiation absorbed at a given energy.

Wastewater Characterization

Testing wastewater characteristics based on Heavy Metal Ions (Pb) values by ICP - OES method.

RESULTS AND DISCUSSION

SEM-EDX from Activated Carbon

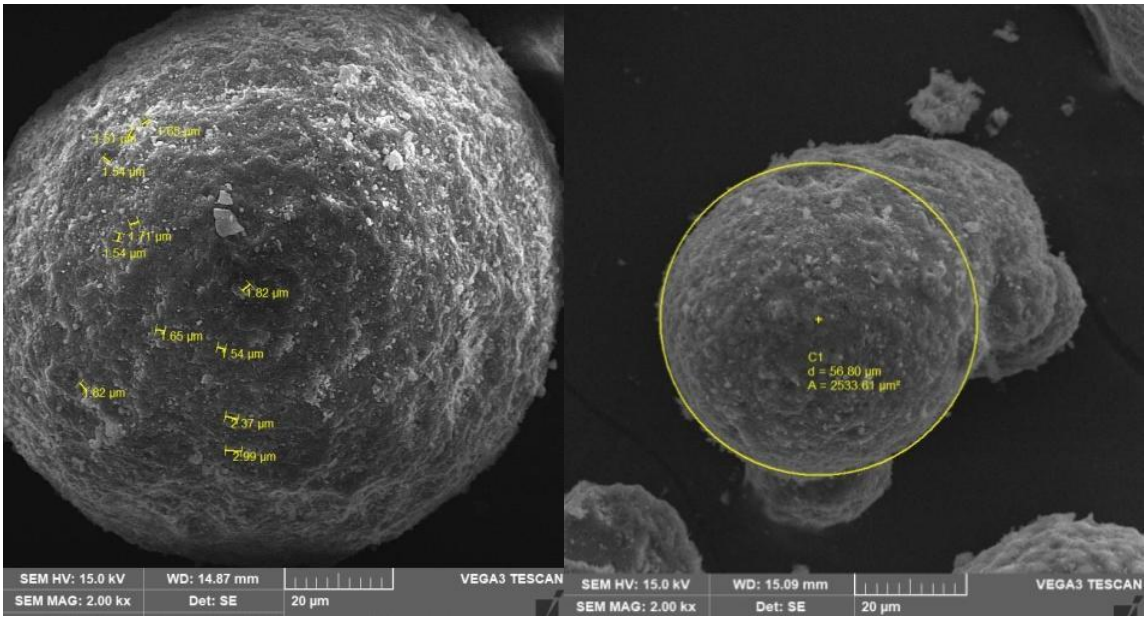

Fig. 1. SEM Figure of Activated carbon at 3000x magnification (Pore Size and Surface Area Characteristics of activated carbon)

It can be seen in Fig. 1 that the results of characterization of activated carbon using SEM can be seen, it can be seen that the largest adsorbent pore size is 2.99µm. Diameter 56.80 and surface area 2533.61 µmµm^2.

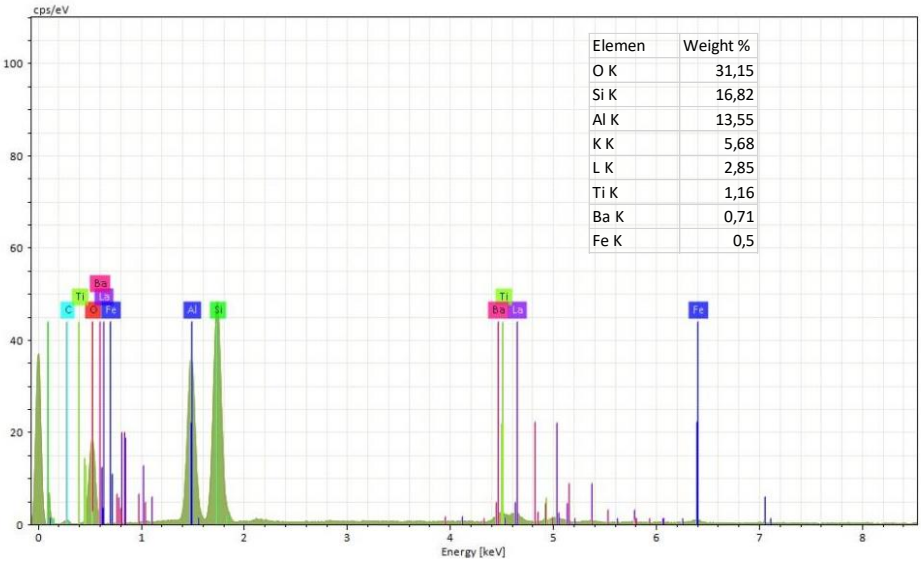

Fig. 2 EDX spectrum activated carbon

FiG. 2. Shows EDX spectrum Used catalyst-based activated carbon. From the picture, we can see the activated carbon silica/alumina ratio of 1.24. Activated carbon containing silicon dioxide and aluminum oxide can increase the adsorption rate [7] . This is because negatively charged alumina silicate can absorb water including heavy metals between its layers [8][9]

EDX analysis can also determine the content contained in used catalyst-based activated carbon. It can be seen that activated carbon has a mass content of Oxygen 31.15%, Silicon 16.82%, Aluminum 13.55%, Carbon 5.68%, Lanthanum 2.85%, Titanium 1.16%, Barium 0.71% and Iron 0.50%.

***Fourier Transform Infrared* (FTIR)**

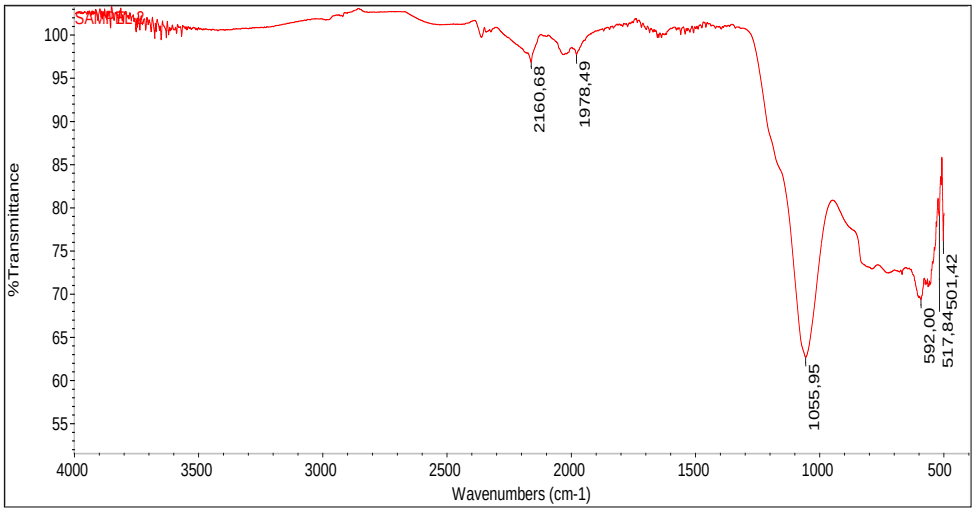

Fig. 3 FTIR Characteristic Graph

In Fig. 3 can be seen the results of characterization with FTIR obtained The first peak position is at wavenumber 2160.68 / cm showing weak but sharp absorption which means the presence of triple compounds C ≡ C, the second peak at wavenumber 1978.49 / cm which means the presence of double compounds C = O, the third peak at 1055.95 / cm which means the presence of compounds C - O, the fourth peak at 592.00/cm, the fifth peak at 517.84/cm and the sixth peak at 501.42/cm are C – H compounds.

Wastewater Characterization

Results of Tofu Industry Liquid Waste Analysis Without Treatment

Table 1 Untreated Analysis Test Results

No	Parameters	Unit	Result

1	Lead (Pb)	mg/L	0,03

The results of the analysis of tofu liquid waste with treatment

Table 2 Test Results With Maceration and Stirring Treatment on Mass Variation

No.	Weight of activated carbon (gr)	Wastewater Volume (ml)	Lead (Pb) with Matching	Stirring Time (minutes)	RESULT Lead (Pb)
1	5	100	0,0291	60	0,0172
2	10		0,0227		0,0146
3	15		0,0243		0,0169
4	20		0,0204		0,0132
5	25		0,0259		0,0101

Table 3 Test Results With Maceration and Stirring Treatment at Time Variation

No.	Weight of activated carbon (gr)	Wastewater Volume (ml)	Lead (Pb) with Matching	Stirring Time (minutes)	RESULT Lead (Pb)
1	10	100	0,0208	30	0,0153
2	10		0,0225	60	0,0101
3	10		0,0198	90	0,0136
4	10		0,0177	120	0,0209
5	10		0,0156	150	0,0175

Effect of RCC Mass Activated Maceration and Stirring Treatment on Tofu Industrial Liquid Waste on Decreasing Lead Concentration (Pb)

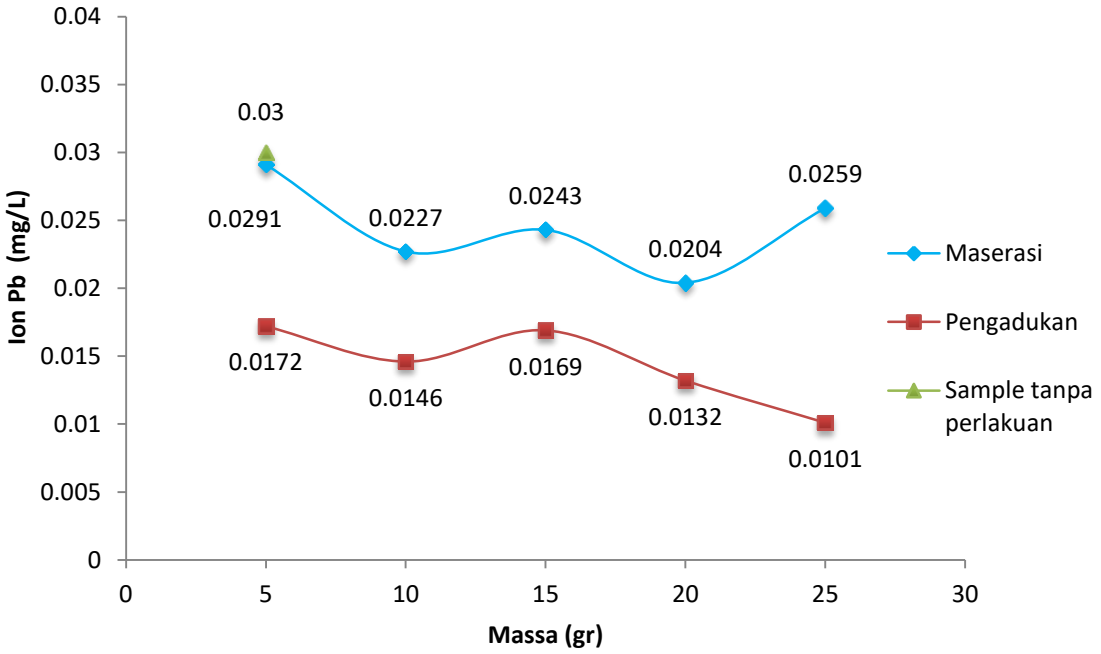

Fig. 4. Effect of Mass on Optimum Mass Maceration and Stirring Treatment Decreased Pb Concentration

The concentration of Pb value of Tofu Industrial Liquid Waste before undergoing treatment with activated activated catalyst-based activated carbon is 0.03 mg / L. Under operating conditions with a contact time of 60 minutes, the RCC mass of 10 gr with maceration treatment obtained a Pb reduction value of 0.0227 mg / L. While with stirring treatment with a mass of 25 gr obtained a Pb decrease value of 0.0101 mg / L.

This difference is because the adsorbent mass and treatment affect the adsorption process carried out. The ability of the adsorbent increases as the mass of the adsorbent increases. This happens because with the addition of the number of adsorbents, there is an increase in the active side contained on the surface of the adsorbent. However, under certain conditions, the percentage of absorption tends to decrease due to saturation of the adsorbent.

The difference in treatment between maceration and stirring also affects the adsorption process. In maceration, the adsorbent is only left in tofu liquid waste, so that the contact process between the adsorbent and tofu liquid waste does not run optimally which results in the adsorption process not running effectively. While in stirring, adsorbents and liquid waste know that they can make optimal contact because they are given stirring. Stirring is one of the factors that affect the adsorption process.

The Effect of Activated Activated Carbon, Maceration and Stirring Treatment on Tofu Liquid Waste on Reducing Lead Concentration (Pb)

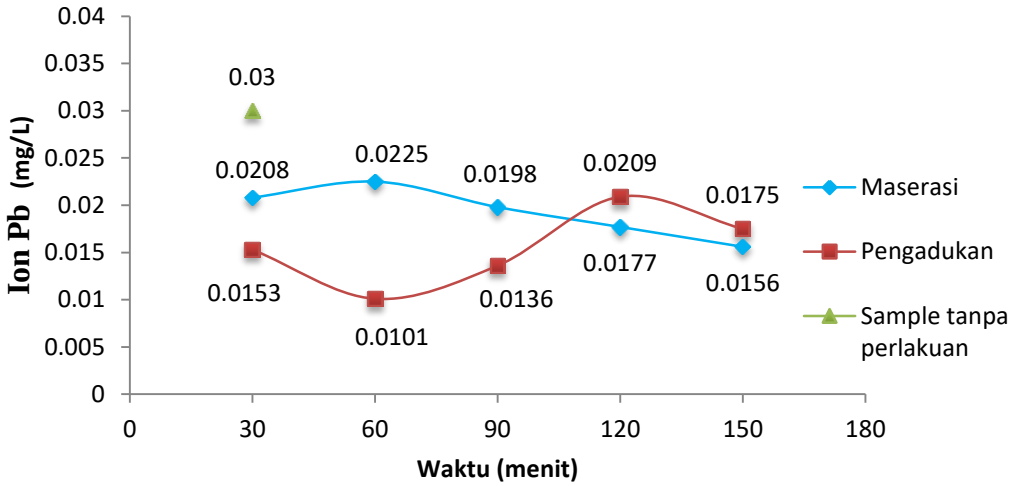

Fig. 5 Graph of the Effect of Time on the Optimum Mass of Maceration Treatment and Stirring Decreased Pb Concentration

It can be seen that the concentration of Pb values with Maceration treatment has the highest / best decrease occurs at 150 minutes with a value of 0.0156 mg / L while for the Stirring treatment the highest / best decrease occurs at 60 minutes with a value of 0.0101 mg / L.

It can be known that the contact time between the adsorbent and the adsorbate affects the adsorption. Contact time affects adsorption because contact time is the length of time the adsorbent and the adsorbate will make contact. Contact time will affect the amount of adsorbate absorbed. This is because each adsorbent has different adsorption capabilities. The longer the contact time between the adsorbent and the adsorban, the more adsorbate will be absorbed but it does not rule out the possibility that if the contact time between the adsorbent and the adsorbate is too long, it will result in a decrease in the ability of the adsorbent due to the saturation of the adsorbent in adsorption.

Contact time affects the amount of adsorbate absorbed, due to differences in the ability of adsorbents to absorb adsorbate differently. Equilibrium conditions will be reached at a time of no more than 150 minutes, after which time the amount of adsorbate absorbed does not significantly change with time.

The decrease in Lead concentration (Pb) occurs because the catalyst waste used is a type of crystalline zeolite that can bind cations because it is negative, for Alumina atomic elements that depend on activated carbon have a charge of 3+ while silica has atomic elements 4+. The existence of this alumina will cause the overall activated carbon based on this used catalyst to have a negative charge, this negative charge

is what causes activated carbon to be able to bind cations in the form of heavy metals which in this study are in the form of Lead (Pb) such as lead which is dangerous in the content of tofu liquid waste

Conclusion

From the results of the study, it is known that the effect of variations in the addition of activated carbon in maceration treatment with liquid waste knows that optimal conditions are obtained at a mass of 10 gr, while for the Stirring treatment, optimal conditions are obtained at a mass of 25 gr. The effect of time conditions on maceration treatment obtained an optimum time of 150 minutes while for the Stirring treatment obtained a stirring time of 60 minutes and a mass of activated carbon of 25 grams with a decrease in Pb ions by 66.33%

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BIBLIOGRAPHY

1. S. Subekti, "PROCESSING TOFU LIQUID WASTE INTO BIOGAS AS AN ALTERNATIVE FUEL," 2011. [Online]. Available: https://www.publikasiilmiah.unwahas.ac.id/index.php/PROSIDING_SNST_FT/article/view/241
2. S. Liang *et al.*, "CONCENTRATIONS OF HEAVY METALS IN UNTREATED PRODUCED WATER FROM A CRUDE OIL PRODUCTION PLATFORM IN NIGER-DELTA, NIGERIA ERAKHRUMEN," *Proc. Natl. Acad. Sci.*, Vol. 3, No. 1, pp. 1–15, 2015, [Online]. Available: http://dx.doi.org/10.1016/j.bpj.2015.06.056%0Ahttps://academic.oup.com/bioinformatics/article-abstract/34/13/2201/4852827%0Ainternal-pdf://semisupervised-3254828305/semisupervised.ppt%0Ahttp://dx.doi.org/10.1016/j.str.2013.02.005%0Ahttp://dx.doi.org/10.10
3. W. W. Nandari, A. Utami, E. Yogafanny, and M. T. Kristiati, "Produced Water Treatment with Bioreactor Membrane in Wonocolo Mining Area," *Exergy*, Vol. 15, No. 2, p. 34, 2018, DOI: 10.31315/E.V15i2.2384.
4. X. Wang, J.:Guo, "Adsorption isotherm: Classification, physical meaning, application and solving method.," *Chemosphere*, Vol. 258, p. 127279, 2020.
5. M. Gao, X.; Fup, C.; Hao, J.; Zhao, Z.; Long, H.; Li, "Adsorption of heavy metal ions by sodium alginate-based adsorpbent- A review and new perspectives," *Int. J. Biol. Macromol*, Vol. 164, pp. 4423–4434, 2020.
6. G. Ray, S.; Das *Chapter 12- Adsorption. In process Equipment and Plant design*. 2020.
7. X. L. and P. L. G. Lu, "Reactivation of spent FCC catalyst by mixed acid leaching for efficient catalytic cracking," *J. Ind. Eng. Chem*, Vol. 92, pp. 236–242.
8. A. G. A. Amari, H. Gannouni, M. I. Khan, M. K. Almesfer, A. M. Elkhaleefa, "Effect of structure and chemical activation on the adsorption properties of green clay minerals for the removal of cationic dye," *Appl. Sci.*, no. 8 (11), p. DOI: 10. 3390/app8112302., 2018.
9. M. K. Uddin, "review on the adsorption of heavy metals by clay minerals, with special focus on the past decade," *Chem. Eng. J.*, No. 308, pp. 438– 462. doi: 10.1016/j.cej.2016.09.029, 2017.