

Reducing Pollutant Levels of Industrial Wastewater using Activated Carbon Based on Used Oil Cracking Catalysts

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Abstract

Treatment of wastewater generated from petroleum exploration is essential to reduce pollutant levels and protect living organisms and the environment. Wastewater produced from the petroleum exploration process is called produced water. Produced water may account for up to 80% of total liquid production and cannot be discharged or reused directly because it contains pollutants such as total dissolved solids (TDS), iron (Fe), and barium (Ba). This study used activated carbon based on catalysts used for petroleum cracking containing Silica/alumina elements as adsorbents to reduce total dissolved solids (TDS) levels and harmful metal content such as Fe and barium. This study aims to determine the effect of variations in adsorbent mass (5, 10, 15, 20, 25 grams) and stirring time (30, 60, 90, 120, 150 minutes) on the decrease in TDS, Fe, and Ba metals. The optimal treatment condition was achieved using a 90-minute stirring period and 15 gram of activated carbon. Under these conditions, Fe and Ba decreased by 79.47% and 62.44%, respectively, and their final concentrations met the stated quality standards. The pH value remained within the acceptable range of 7.12 to 8.10. TDS decreased by 54.67% to 6,120 mg/L, but remained above the stated limit and therefore requires further polishing treatment.

Keywords: *Activated carbon; used catalyst; pollutant; adsorbent; industrial wastewater; produced water*

INTRODUCTION

Oil is Indonesia's main export and is used as fuel and raw material for the petrochemical industry. Oil trading activities play an important role in national economic development. The Prabumulih oil and gas field, operated by PT Pertamina EP, is among the largest oil and gas production areas in Indonesia, comprising 201 production wells and 73 injection wells ([Said et al., 2021](#)). A number of oil and gas wells in Indonesia have reached a mature stage, requiring greater volumes of water

injection to sustain oil production. The volume of produced water generated each day is very large, reaching an average of approximately 80% of the total fluid production in each well. After the oil and water separation stage, the separated water is referred to as produced water and is mostly reinjected into the well. However, when reinjection is not technically feasible because of well-related operational problems, the produced water must be released into the environment. Before being discharged into the environment, produced water needs to be treated first to meet quality standards in accordance with the Regulation of the Minister of Environment of the Republic of Indonesia Number 5 of 2014 concerning Wastewater Quality Standards.

Produced water is water resulting from oil and gas production activities. Unlike conventional water, produced water contains potentially hazardous chemicals and various constituents originating from oil and gas formations. The volume of produced water generally increases as oil or gas fields age, although appropriate treatment and the development of new oil or gas fields may reduce the amount generated. Produced water can come from salt water and formation water. Produced water cannot be discharged and utilized directly because it contains several parameters such as COD, oils and fats, TDS, pH, Fe, and Barium. High Fe concentrations may deteriorate water quality, cause discoloration, and promote sediment formation. Excessive Ba may pose risks to aquatic organisms and contribute to scale formation, while high TDS concentrations can increase salinity and reduce the suitability of water for discharge or reuse. The utilization of produced water and the disposal of produced water that has not been treated and contains many hazardous materials can disrupt the balance of the environment.

Therefore, better treatment is needed by treating produced water with the adsorption method using activated carbon based on used catalysts. Processing using adsorption technology uses adhesive carbon as an absorbent of pollutants contained in industrial wastewater. Adsorption technology uses porous carbon materials as adsorbents to retain pollutants contained in industrial wastewater. With the adsorption method, it is expected that produced water with excellent quality will be produced, which is stable and in accordance with quality standards when the produced water is utilized and discharged into the environment. The treated produced water must meet quality standards according to South Sumatra Governor Regulation No. 8 of 2012.

Produced water can be treated through various technologies, such as adsorption, membrane filtration, ion exchange, electrocoagulation, and reverse

osmosis. Membrane filtration and reverse osmosis can effectively separate suspended and dissolved pollutants, but their application may be limited by membrane fouling, energy requirements, and the production of concentrated reject streams. Ion exchange is effective for removing dissolved ions but requires resin regeneration, while electrocoagulation can remove metals and suspended pollutants but may generate sludge and consume electrical energy. Compared with these technologies, adsorption offers a relatively simple process and enables the utilization of low-cost or waste-derived materials as adsorbents ([Pinto, P. X., Al-Abed, S. R., Balz, D. A., Butler, B. A., Landy, R. B., & Smith, 2016](#); [Said et al., 2021](#); [Novira et al., 2022](#)).

Various approaches have been applied to treat contaminated water, including boiling, pasteurization, chlorination, disinfection through flocculation, ultraviolet (UV) irradiation, and biosand filtration ([Pinto, P. X., Al-Abed, S. R., Balz, D. A., Butler, B. A., Landy, R. B., & Smith, 2016](#)). Activated carbon represents a promising and appropriate alternative for the small-scale treatment of water intended for household consumption ([Furqoni et al., 2015](#)). In addition, this material has good thermal stability, resistance to biological or microbial degradation, various potential raw materials, and relatively low cost ([Ma'ruf et al., 2015](#)).

In research on making activated carbon, the composition of raw material content needs to be known in order to obtain activated carbon that is able to carry out the absorption process.

Spent catalyst is a residual material generated from the petroleum cracking process. Spent catalyst waste generated by PT Pertamina RU III is a regularly structured crystalline zeolitic material containing oxide compounds, potassium, magnesium, and rare-earth elements, including lanthanum and cerium. In refineries equipped with a residue catalytic cracking (RCC) unit, this catalyst is used to promote and accelerate reactions that produce major products such as LPG, propylene, polygasoline, naphtha, LCO, and decant oil. The use of used catalysts as the main material in making activated carbon because it requires large costs to be regenerated so that it becomes useless and has the potential to become waste. Therefore, its utilization as an adsorbent may reduce industrial waste while providing an alternative material for produced water treatment. The used catalyst used comes from Pertamina RU III Plaju, which has gone through a petroleum cracking process. This used catalyst is a type containing oxides of silica and alumina, as well as other elements including SiO₂ 38.27%, Al₂O₃ 51.37%, Fe₂O₃ 0.527%, TiO₂ 0.461%, CaO 0.117%, and MgO

0.145% ([Ermawati et al., 2016](#)). The results of research analysis conducted by Ermawati in 2016 showed that the main component of used catalysts is almost the same as zeolite, so that from this used catalyst can be used as the main material for making activated carbon. The high silica and alumina contents indicate the potential of spent cracking catalyst as an adsorbent for dissolved metals and other pollutants in produced water.

The adsorption process generally occurs on the surface of the adsorbent, so that the surface area of the adsorbent becomes a determining parameter for the success of the process. Adsorbents are generally of porous materials or particles so that they have a large surface area that correlates with high adsorption capacity. The inner walls of pores on porous particles can increase the internal surface area. Highly porous particles can have an internal surface area between 102 m²/g and 103 m²/g, whereas the external surface area is usually below 1 m²/g (Worch, 2012). Internal surface area for *Peanut Hull Carbon* (PHC) has been found to be 485 m²/g. A high internal surface area is required to provide sufficient adsorption sites and support an effective separation or purification process. Adsorbent materials may exhibit internal surface areas ranging from 100 to 3,000 m²/g, although values between 300 and 1,200 m²/g are more commonly applied, with pore diameters typically ranging from 0.0003 to 0.02 µm ([Gul Zaman et al., 2021](#)).

Experimental Section

Materials

Industrial wastewater samples (produced water) were obtained from one of the oil and gas companies located in South Sumatra Province. Spent catalyst-derived activated carbon was obtained from a petroleum processing plant located in South Sumatra. The material was a residual catalyst from the petroleum cracking process that was no longer reused in the processing unit. The spent cracking catalyst had a particle size of 100 mesh. The spent catalyst-based activated carbon used in this study had a silica/alumina ratio of 1.26. The surface properties and morphology of the activated carbon were examined using a Hitachi FlexSEM 1000 scanning electron microscope coupled with energy-dispersive X-ray spectroscopy.

Solution Preparation

The spent catalyst used as the activated carbon material was initially cleaned to remove adhering impurities. After cleaning, the regeneration and activation processes were conducted by calcination in a furnace at 700°C for 1 hour. Following activation, the catalyst was cooled in a desiccator and kept there for 24 hours to preserve its stability. The material was then sieved to obtain a particle size of 100 mesh. The industrial wastewater sample was collected from the oil company site in a 500 mL storage container. Before the adsorption process, the produced water was filtered using a 0.1 µm polypropylene filter to remove suspended impurities and solid particles.

METHOD

In this study, produced-water treatment was conducted through two sequential stages. The first stage is the filtration process using a polypropylene filter. The second stage is the adsorption process using used-year catalyst-based activated carbon. Prior to adsorption, the spent catalyst was purified to eliminate adhering impurities, thermally treated at 700°C for 1 hour, cooled and maintained in a desiccator for 24 hours, and subsequently sieved to obtain a particle size of 100 mesh.

The Fe, Ba, TDS, and pH values of the produced water were analyzed before and after the adsorption process. Iron and barium concentrations were quantified using a Shimadzu AA-7000 atomic absorption spectrophotometer, whereas TDS and pH were determined using a Horiba LAQUA PC210-K TDS meter and a Horiba LAQUA pH meter, respectively. The removal efficiencies of Fe, Ba, and TDS were calculated using the following equation:

$$Removal (\%) = \frac{(C_{in} - C_{out})}{C_{in}} \times 100\% \quad (1)$$

where C_{in} represents the initial concentration of the sample and C_{out} represents the concentration after treatment.

Determination of the Optimum Mass and Time Ratio

Different adsorbent dosages of 5, 10, 15, 20, and 25 g were introduced into separate 100 mL aliquots of produced water. The suspensions were then agitated at a constant rate with a magnetic stirrer for 30, 60, 90, 120, and 150 minutes. After each contact time, the adsorbent was separated from the filtrate, and the resulting filtrate was analyzed for TDS, pH, Fe, and Ba.

Research Analysis

Characterization of activated carbon based on used catalysts (Adsorbents)

a. Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) uses a focused beam of high-energy electrons to produce signals through interactions with the surface of a solid specimen. These signals provide detailed information regarding the material's surface morphology.

b. Energy Dispersive X-Ray (EDX)

Energy-dispersive X-ray spectroscopy (EDX) was employed to determine the elemental composition and chemical characteristics of the sample by analyzing element-specific X-ray emissions generated during electron-beam interaction. The constituent elements were identified from the characteristic signals recorded at selected points on the sample surface.

c. Fourier Transform Infrared (FTIR)

Fourier transform infrared (FTIR) spectroscopy identifies chemical compounds by analyzing molecular vibrations. The resulting spectrum is obtained by transmitting infrared radiation through the sample and recording its absorption at specific wavenumbers.

Characterization of Produced Water

The produced-water characteristics were evaluated based on the measured TDS, pH, Fe, and Ba values.



Figure 1. Used catalyst-based activated carbon a) before activation b) after activation

RESULTS AND DISCUSSION

SEM-EDX, FTIR from Used Catalyst-Based Activated Carbon

Analyzing the sample's surface, pores, and particle size was carried out by the Scanning Electron Microscopy (SEM) method. Meanwhile, to analyze the elements

characterizations of activated carbon are specifically carried out by EDX Analysis. SEM-EDX provides a complete overview of the elemental composition of activated carbon. The surface morphology and elemental characteristics of the activated carbon before and after activation, as determined by SEM-EDX analysis, are presented in Figures 2 and 3.

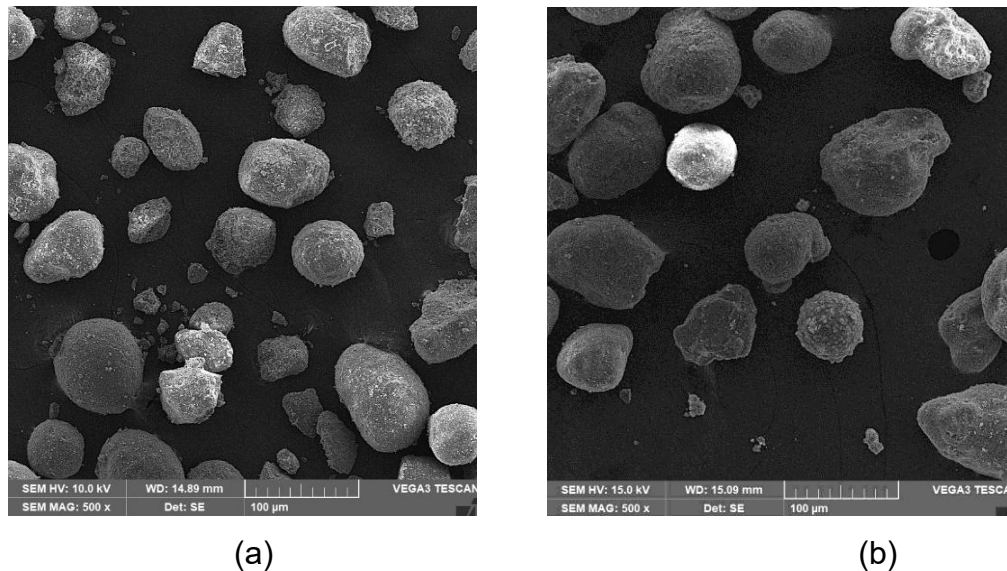
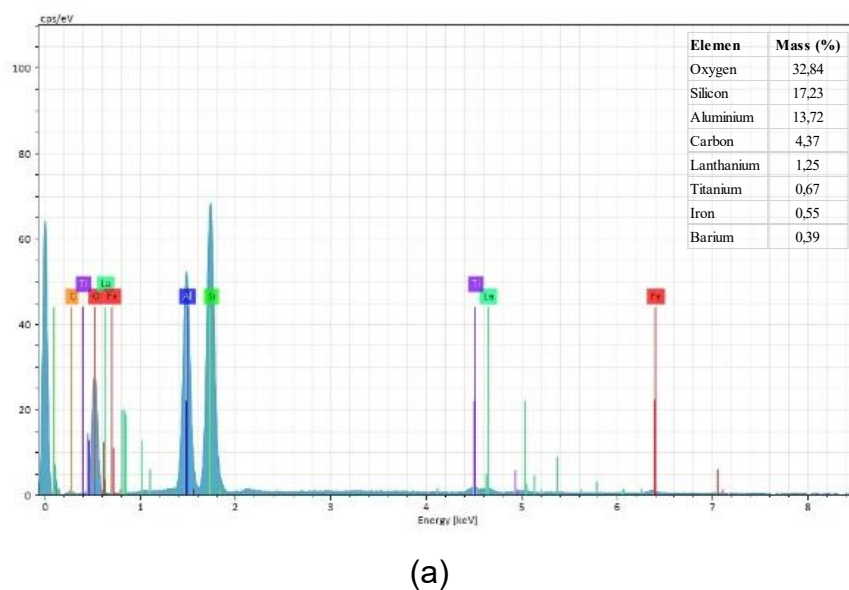
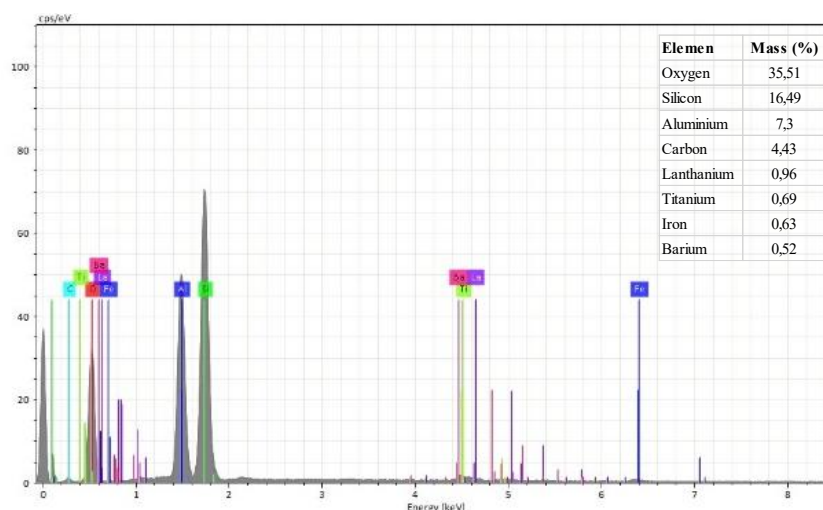


Figure. 2. SEM image of activated carbon Used catalyst-based activated carbon at 500 x magnification (a) before activation (b) after activation

Results of Characterization of Used Catalyst Waste with *Energy Dispersive X-Ray* (EDX/EDS)





(b)

Figure 3. EDX SEM spectrum activated carbon Used catalyst (a) before activation (b) after activation

Figure 3 presents the EDX spectra of the activated carbon before and after the activation process. Following activation, the silica-to-alumina ratio increased from 1.26 to 2.26. This compositional change may contribute to a larger surface area and pore volume, thereby improving the adsorption capacity of the material (Erliyanti et al., 2020). Moreover, the presence of silicon dioxide (SiO_2) and aluminum oxide (Al_2O_3) in clay-based materials has been reported to support enhanced adsorption performance (G. Lu et. al, 2020). Most clay minerals can expand, increasing their interlayer spacing and allowing water molecules and ionic species to be retained. The negatively charged aluminosilicate surfaces within clay layers can also bind water and dissolved heavy-metal ions (A. Amari, H. Gannouni, M. I. Khan, M. K. Almesfer, A. M. Elkhaleefa, 2018).

Table 1. BET Used catalyst-based activated carbon

Characterization	Value
Specific surface area (m ² /g)	62,004
Pore Size (nm)	1,686
Pore Volume (cm ³ /g)	0,100

The specific surface area, pore size, and pore volume of the activated carbon were measured using Brunauer-Emmett-Teller (BET) analysis, as shown in Table 1. According to BET analysis, the activated carbon's pore size is 1.686 nm and its outer surface area is 62.004 m²/g. Activated carbon is categorized into micropores (diameter <2 nm) based on pore size (Amakiri, K. T., Canon, A. R., Marco, M. and

[Dimakisa, 2022; Ngoc Dung, T. T., Phan Thi, L. A., Nam, V. N., Nhan, T. T. and Quang, 2019\).](#)

Characterization Results of Activated Carbon Used catalyst with *Fourier Transform Infrared* (FTIR)

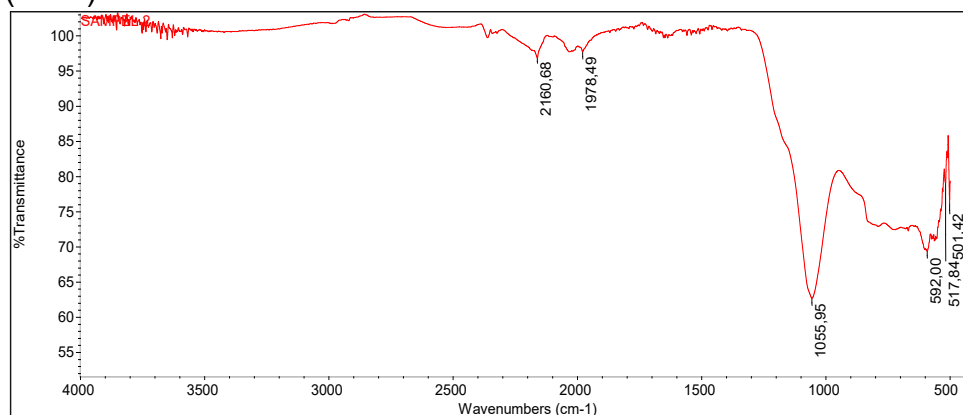


Figure 4. FTIR Characteristic Graph

Figure 4 shows absorption bands at wavenumbers of 2160.68, 1978.49, 1055.95, 592.00, 517.84, and 501.42 cm^{-1} . The asymmetric stretching of Si-O-Si and Si-O-Al connections within the aluminosilicate framework is indicated by the absorption band at 1055.95 cm^{-1} . The bands at 592.00, 517.84, and 501.42 cm^{-1} are associated with Si-O and Al-O framework-bending vibrations. The weak bands at 2160.68 and 1978.49 cm^{-1} may be associated with overtone or combination bands and should not be directly assigned to $\text{C}\equiv\text{C}$ and $\text{C}=\text{O}$ groups without further supporting analysis. These assignments are consistent with the silica and alumina composition identified by EDX.

Characterization of Produced Water

The physical-chemical characterization of produced water is described in Table 2. The pH, TDS, barium, and ferrous metal values in produced water have exceeded the limits set by the Indonesian Minister of Environment Regulation No. 5 of 2012.

Table 2. Characterization of produced water

No	Parameters	Unit	Standard
Ph	-	6-9	7,82
TDS	Mg/L	4000	13500
Iron (Fe)	Mg/L	0,3	0,51
Barium	Mg/l	2	5

Acidity

The initial characteristic of the acidity level of produced water is 7.82. Table 3 shows the acidity value of the filtrate from ceramic adsorbents with different mixed compositions (Adsorbent A to Adsorbent F).

Table 3. Acidity permeates at different stirring times and masses of activated carbon

Mixing time, min	activated carbon mass, grams	Activity
30	5	8,09
	10	7,84
	15	7,83
	20	7,93
	25	8,03
	Average	7,94
60	5	8,01
	10	7,77
	15	7,65
	20	7,81
	25	7,92
	Average	7,83
90	5	8,04
	10	7,58
	15	7,12
	20	7,17
	25	7,18
	Average	7,42
120	5	8,02
	10	7,77
	15	7,82
	20	8,05
	25	8,10
	Average	7,95
150	5	8,08
	10	7,66
	15	7,82
	20	7,58
	25	7,85
	Average	7,80

Table 3 shows that the pH of the treated produced water ranged from 7.12 to 8.10 at stirring times of 30 to 150 minutes and activated carbon masses of 5 to 25 grams. The acidity value of water produced by treatment using activated carbon is still within the permissible limits, which is 6-9. However, there was an increase in the

acidity value of the initial produced water of 7.82, this is due to differences in the ability of alumina and silica to react with bases ([Novira et al., 2022](#)). Alumina oxide is amphoteric because it can react with strong acids and bases. While silica is a non-metallic oxide compound that is covalently bonded and can react with strong bases ([Malakootian, M., Mahvi, A. H., Mansoorian, H. J., & Khanjani, 2018](#)). The acidity value is close to neutral found in activated carbon with 30 minutes of stirring which is 7.84; 7.83; 7.93, stirring time 60 minutes 7.77; 7.65; 7.81; 7.92, 90-minute stirring time is 7.58; 7.12; 7.17; 7.18, stirring time of 120 minutes which is 7.77; 7.82, and stirring time 150 which is 7.66; 7.82; 7.58; 7.85. With a mass of 15 grams of activated carbon and a stirring period of 90 minutes, the acidity value that is closest to neutral is 7.12. Overall, the variations in stirring time and activated carbon mass did not cause the pH of the produced water to exceed the permitted range.

The Effect of Stirring Time and Activated Carbon Mass on the Decrease in Pollutant Value

Iron (Fe) Contained in Produced Water

Iron (Fe) levels in water can serve as a gauge for the presence of heavy metal contamination. The initial Fe concentration of 0.51 mg/L was higher than the allowable limit of 0.30 mg/L, as per Indonesian Minister of Environment Regulation No. 5 of 2014 on wastewater quality requirements. In these rules, water can be said not to be polluted if the maximum value of iron in the produced water is 0.3 mg/L or iron content is ≤ 0.3 mg/L.

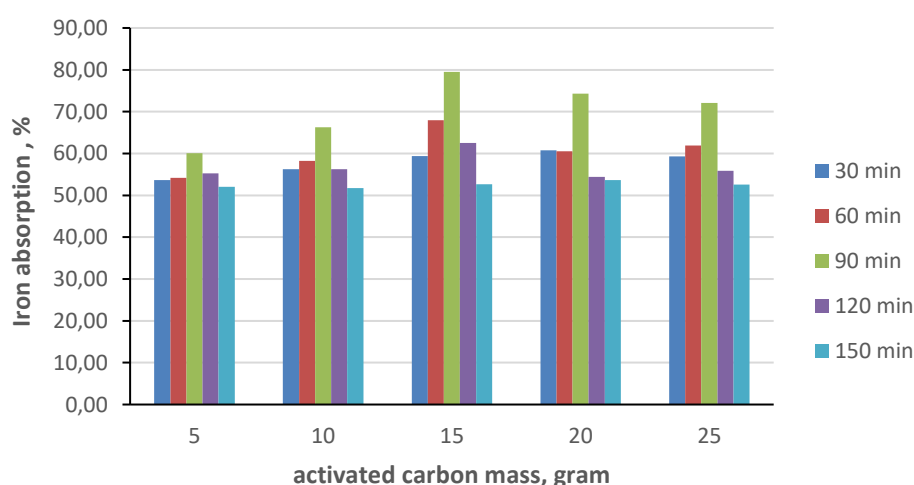


Figure 5. Iron removal by activated carbon

As shown in Figure 5, the optimum condition was achieved at a stirring time of 90 minutes and an activated carbon mass of 15 g, resulting in 79.47% Fe removal and a final Fe concentration of 0.1047 mg/L.

Total Dissolved Solids

It is known that the total dissolved solids level of produced water taken from one of the oil drilling units in South Sumatra has a TDS level of 13,500 mg/l.

Industrial wastewater before the adsorption process has previously been carried out a filtration process using a polypropylene filter to separate solid particles carried when the wastewater is taken.

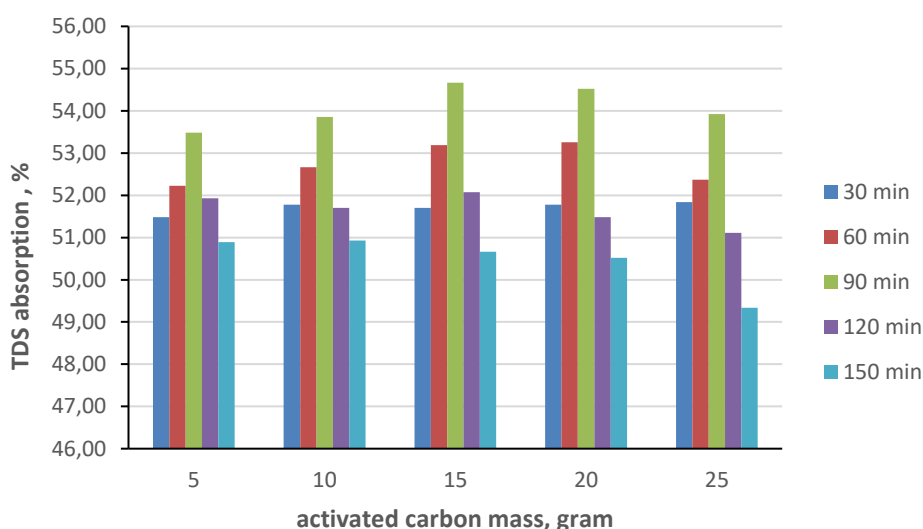


Figure 6. TDS removal of produced water by activated carbon

Total Dissolved Solids (TDS) refers to the concentration of dissolved organic and inorganic substances present in water. The high TDS content in the resulting water can allow fouling of activated carbon so as to reduce adsorption results. After 90 minutes of stirring and 15 grams of activated carbon, the TDS value decreased to 7,380 mg/L. TDS absorption of 54.67% (TDS content in wastewater of 6,120 mg/L).

Barium (Ba) Contained in Produced Water

Barium is the most common metal ion and is found abundantly from industrial waste (produced water). The Republic of Indonesia's Ministry of Environment and Forestry's Regulation No. 5 of 2014 states that the maximum barium ion concentration in effluent from oil and gas enterprises is 2 mg/L. According to the findings of the

preliminary wastewater analysis, the barium ion level is 5 mg/L. The maximum amount permitted by government restrictions is exceeded by this value.

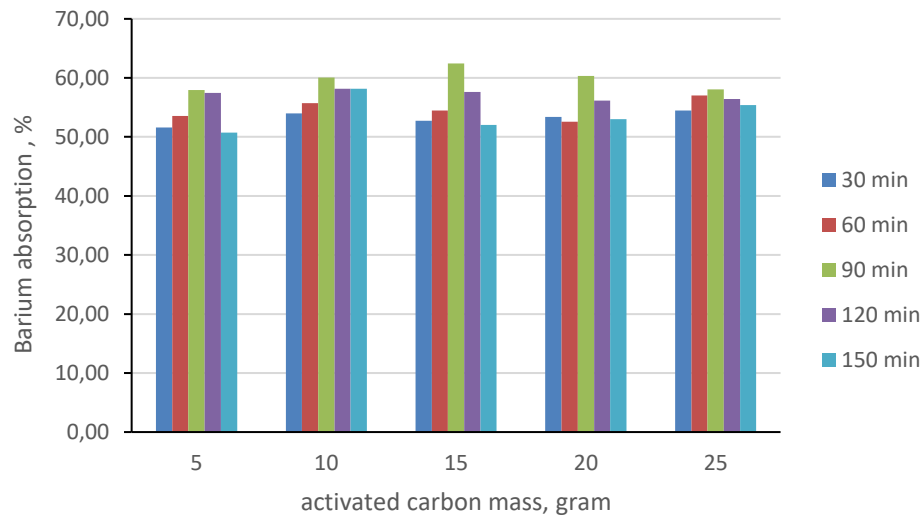


Figure 7. Barium removal of produced water by activated carbon

With a mass of 15 grams of activated carbon and a percentage of barium absorption of 62.44%, Figure 7 demonstrates that the lowest barium level in wastewater following the adsorption process, 1.88 mg/L, was achieved after 90 minutes of stirring. Only a 90-minute stirring time variation can lower the barium ion content in wastewater (activated carbon weight of 15 and 20 grams); other conditions (30, 60, 120, and 150 minutes) cannot lower barium concentrations to levels specified by Republic of Indonesia regulation No. 5 of 2014's Ministry of Environment and Forestry.

The following formula was used to get the adsorption capacity:

$$q = \frac{(C_0 - C_e)V}{m}$$

where q is the adsorption capacity in mg/g, C_0 is the initial pollutant concentration in mg/L, C_e is the final pollutant concentration in mg/L, V is the sample volume of 0.1 L, and m is the mass of the adsorbent in grams.

Table 4. Pollutant removal at the optimum adsorption condition

Parameter	Initial concentration (mg/L)	Final concentration (mg/L)	Removal efficiency (%)	Adsorption capacity (mg/g)
Fe	0.51	0.1047	79.47	0.0027

Ba	5.00	1.88	62.44	0.0208
TDS	13,500	6,120	54.67	49.20

Table 4 summarizes the initial and final pollutant concentrations obtained under the optimum adsorption condition of 90 minutes and 15 grams of activated carbon. The calculated adsorption capacities for Fe, Ba, and TDS were 0.0027, 0.0208, and 49.20 mg/g, respectively.

Fe and Ba can be eliminated from the silica-alumina surface by surface complexation, ion exchange, and electrostatic attraction. Negatively charged sites that interact with dissolved Fe and Ba ions can be formed by silanol groups (Si-OH) and aluminum groups (Al-OH). The aluminosilicate framework may also exchange its charge-balancing ions with dissolved metal ions. However, the high concentration of dissolved substances in produced water may cause competition among ions for the available adsorption sites ([A. Amari, H. Gannouni, M. I. Khan, M. K. Almesfer, A. M. Elkhaleefa, 2018](#)).

The condition of 90 minutes and 15 grams was considered optimal because it produced the lowest final concentrations and the highest simultaneous removal efficiencies for Fe, Ba, and TDS. A contact time of 90 minutes provided sufficient interaction between the pollutants and the available adsorption sites. Longer stirring times did not improve pollutant removal, possibly because the readily available sites had become saturated or because pore blockage, ionic competition, and partial desorption occurred. An activated carbon mass of 15 grams provided sufficient adsorption sites, whereas higher masses did not improve removal, possibly because of particle agglomeration and overlapping adsorption surfaces.

CONCLUSION

The study leads to the following conclusion.

- Spent catalysts from crude oil cracking processes can be utilized as adsorbents under appropriate operating conditions to reduce pollutant concentrations in industrial wastewater, particularly produced water.
- The acidity value of the produced water after the treatment process ranged from 7.12 to 8.10. At the optimum condition of 90 minutes and 15 grams of activated

carbon, the pH value was 7.12 and remained within the permitted range of 6 to 9.

- At the optimum condition, the Fe concentration decreased by 79.47%, from 0.51 to 0.1047 mg/L, while the Ba concentration decreased by 62.44%, from 5.00 to 1.88 mg/L. These final concentrations met the stated wastewater quality standards of 0.3 mg/L for Fe and 2 mg/L for Ba.
- The TDS concentration decreased by 54.67%, from 13,500 to 6,120 mg/L. However, the final TDS concentration remained above the stated limit of 4,000 mg/L. Therefore, adsorption using spent catalyst-based activated carbon was effective in reducing TDS but was insufficient as a single treatment process.
- The optimum stirring time and activated carbon mass for reducing pollutant levels in produced water were 90 minutes and 15 grams, respectively. Additional treatment is required to further reduce TDS before discharge or reuse. The adsorption process may be integrated with reverse osmosis, ion exchange, membrane filtration, or another polishing process to achieve the required effluent quality.

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