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Activated Carbon from Coffee (*Coffea Arabica L.*) Husk Impregnated with $ZnCl_2$ dan $CaCl_2$ for the Adsorption of Tofu Wastewater

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Abstract. *Tofu wastewater effluent contained high value of BOD, COD, and suspended solids which needed treatment before disposed to environment. The research aims to study the adsorption of BOD and COD* in *tofu wastewater using chemically impregnated carbon derived from coffee husks (Coffea Arabica L.). The Arabica coffee husk carbon (ACHC) activated with $ZnCl_2$ 0.1 N and $CaCl_2$ 0.1N for 3 hours. Carbon characterization compared with Indonesian standard of SNI No.06-3730-1995, and the surface area was examined by Fourier transform infrared spectroscopy (FTIR). The results showed that the moisture and ash content characteristics has met the Indonesian standard, but the iodine adsorption capacity was decreased with longer activation time. The highest iodine adsorption recorded at 888.37 mg/gr adsorbent using ZnCl2 0.1 N at 1 h activated time. The BOD and COD removal efficiency were 88% and 25%, for ACHC with $ZnCl_2$ 0.1N; Meanwhile carbon with $CaCl_2$ 0.1N has a removal efficiency 66% and 24% for BOD and COD, respectively. In conclusion, the variation of chemical activators and the longer contacted time gave significant effect on the removal of pH, BOD and COD from tofu wastewater. To obtain optimum COD adsorption efficiency in wastewater treatment, other factors such as the impregnation ratio, the activators concentration, the mixing time, and its combination method were needed further investigation.*

Keywords: *arabica coffee husks, activated carbon, chemical activators, contact time, tofu wastewater*

1. Introduction

The tofu industry is mainly a household-scale industry that produces wastewater with a high organic content (Astuti et al, 2007). Tofu wastewater containing COD, BOD_5 and Nitrite which has potency in polluted water if it were not well treated (Sartika, 2019). Tofu wastewater contains 9% protein, 0.69% fat, and 0.05% carbohydrates. The remaining wastewater still contained nutritional such as protein which allows protease-producing microorganisms to grow (Fatoni, 2008). Each quintal of soybeans for tofu processing will produce about 1.5 – 3.0 m3 of wastewater. This waste still contains several organic components such as protein and fat which can cause eutrophication, and an unpleasant odor when discharged directly into the waters. (Damayanti, 2004). Several methods have been applied to reduce tofu wastewater pollution level, included coagulation, oxidation, degradation with UV light, adsorption, and biological process using microorganisms (Suyata, 2009). According to Lahti et al. (2017), activated carbons are broadly used as adsorbents or as supported materials in different industrial processes, because it easily prepared from residual or waste biomass materials. Bio sorbent prepared from agricultural waste is a useful material due to its efficiency, low cost, environment friendliness, and availability in large quantity.

The most significant factors affecting the pore's structure of super activated carbon

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were the activation temperature, the activation time, and the impregnation ratio (Li et al., 2019). Some studies have examined the use of chemical activators in the processing of activated carbon derived from coffee biomass waste. Rasdiansyah (2014) characterized the effect of activation time and carbonization temperatures of 400 ^{0}C, 500 ^{0}C and 600 ^{0}C on the iodine adsorption capacity using activated carbon impregnated with ZnCl2 derived from coffee spent powder. Raudah (2014) has made activated carbon from coffee hard skin which activated with HCl 0.1 N and it was able to remove wastewater cyanide of 84.13%. Phuong et al., (2021) found that variation of temperature of pyrolysis (300, 450 and 600 0C) of coffee husk biochar had significant effect on pH, pHpzc and mmolOH- of water treated, while the highest NH4+ removal was obtained at lower pyrolysis temperature. Other research by Matilda et al., (2016) showed that activation of the Ulin wood carbon increased the surface area of 53,7% and improved the pH and BOD quality from tofu wastewater effluent.

Subroto (2017) claimed that carbon from coffee husks has a high calorific value, low moisture content, and a low sulfur content. Therefore, coffee husk, which are mostly produced from smallholder plantations have the potential to be used as raw material for activated carbon. The highest producer of Arabica coffee in Aceh Province are Central of Aceh and Bener Meriah Districts, in Gayo Highland area. The hulling process of fresh coffee seed produced coffee husk or “kulit tanduk” which mainly dumped or burnt nearby the warehouse. Only some of the best quality coffee husk used as cascara tea nowadays. Data from Indonesian Central Bureau (BPS) (2021) Coffee production in Indonesia has reached 643.857 tonne. Martauli (2018) reported that Arabica coffee is commonly planted by farmers in Aceh, North Sumatra, South Sulawesi, Bali and Flores.

Based on those findings, it is necessary to characterize the effect of chemical activators impregnation for the removal and the adsorption of tofu wastewater parameters, as the selection of the type of activator will affect the quality of the activated carbon produced. Chemical activators commonly used were $CaCl_2$, $MgCl_2$, $ZnCl_2$, NaOH, KOH, HNO_3, H_2SO_4, and HCl. But in this study, we focused on the activation of $CaCl_2$ 0.1 N and $ZnCl_2$ 0.1 N for 3 hours to acknowledge the characterization of AHAC carbon using Indonesian National Standard (SNI) and its functional groups by FTIR. It also aims to study the effect of variated contacted time of 1, 3 and 5 hours for removing the COD and BOD parameters. Furthermore, the adsorption of AHAC into tofu wastewater was assumed to meet the Indonesian industrial wastewater effluent standards before being discharged into water body.

2. Method

2.1 Materials and Equipment

Equipments: oven *Memert*; furnace *Spektronosifik*; blender *Miyako*; sieve mesh; Beaker volume 250 ml *Pyrex;* erlenmeyer *pyrex*; watch glass *pyrex*; pH meter *Hanna*; *magnetic stirrer*; *hot plate wisd*; DO Meter *Hanna*; COD analyzer, water sampler.

Materials: Arabica coffee husk from Gayo Plantation – Aceh; Tofu wastewater from Home Industry Mawar Banda Aceh; Aquadest; Chemical Activators $CaCl_2$ 0,1 N and $ZnCl_2$ 0,1 N were purchased commercially from Merck.

2.2 Research Method

Activated Carbon Preparation

Arabica coffee husk waste collected from fresh coffee hulling and milling in Gayo Highland, Bener Meriah, Aceh Province. Wet coffee husk was washed thoroughly with

Proceeding of ICMR 6(2), 279-289 DOI: https://doi.org/10.32672/picmr.v6i2.1273

aquadest and sun dried for 3 days. Dried coffee husk was oven at 105^{0}C for 3 hours until constant weigh was reached. To produce activated carbon from Arabica coffee husk, the oven dried coffee husk subjected to furnace at temperature 400^{0}C for 2 hours. The carbon produced were blend and sieve using 60 sieve mesh. The impregnation process to activate the carbon was done chemically using $ZnCl_2$ 0,1N and $CaCl_2$ 0,1N with variated contacted time (1, 3 dan 5 hours). The excess of $ZnCl_2$ and $CaCl_2$ in activated carbon were washed throughly with aquadest until neutral pH was reached. The carbon residue dried in oven for 2 hours at 200^{0}C to reduce the moisture content.

Characteristics of activated carbon Using Indonesian Standard (SNI) No. 06- 3730-1995

The activated charcoal ACHC was analysis procedure refers to the Indonesian National Standard 06-3730-1995 for technical activated charcoal which includes water content, ash content, iodine absorption capacity. Characteristic analysis was carried out before and after activation with $ZnCl_2$ 0.1N and $CaCl_2$ 0.1N.

2.3 Analysis of the Adsorbent Functional Group Using FTIR Method

Determination of the functional groups found on the surface of activated carbon pores was carried out using the FTIR (Fourier Transform Infra Red) test. FTIR is an emission absorption method that observes the interaction of molecules with electromagnetic radiation in the wavelength region of 400 - 4000 nm. The research used activated carbon samples from porous Arabica Gayo coffee husk with 3 repetitions on a total of 3 test. FTIR analysis done for the carbon activated with $ZnCl_2$ 0.1N, $CaCl_2$ 0.1N and after contact with tofu wastewater.

2.4 Analysis on Adsorbent Contacted Time

The pH, COD and BOD contents used came from liquid waste samples from the tofu industry. Determination of contact time was carried out by taking 1000 ml of tofu liquid waste and putting it into a different 1,500 ml Erlenmeyer flask, then contacting it with an adsorbent at a dose of 10 gr/L, with variations in contact time of 1, 3 and 5 hours. The solution was then stirred with a magnetic stirrer at a constant speed. Each tofu liquid waste is then filtered and analyzed for the final reduction in pH, BOD and COD. The results of the final reduction analysis were used to analyze the adsorption efficiency.

2.5 *Data Analysis*

Data analysis that carried out included the decrease in pH, COD and BOD parameters before and after contacted with chemically activated coffee peels activated carbon CaCl2 and ZnCl2. pH value on waste samples is carried out based on SNI 6989.59:2008. Meanwhile, COD content was examined based on SNI. 06.6989.15:2004, and BOD was done based on Dwinanto (2009).

3. Results and Discussions

Preparation of Arabica coffee husk carbon (ACHC) were done by drying the coffee husk in the sun for 3 days, then oven at 105 ^{0}C for 1.5 hours until constant weight were gained. The coffee char produced were grinded at 60 mesh and kept in the seal tight container for the whole research. Activated carbon prepared from Gayo coffee husks is shown in Figure 1 below.

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Figure 1. Dried coffee arabica husk (left) and arabica coffee husk carbon (right)

3.1 Characteristics of Arabica Carbon Before and After Chemical Activation

Table 1 described the results of the characteristics of activated carbon before chemical activation and after chemical activation of $ZnCl_2$ 0,1N and $CaCl_2$ 0,1N. The water content at the activation time of 1, 3 and 5 hours all meet SNI standards which is below 15%. The ash content of activated carbon at a contact time of 5 hours is much higher than the good quality standard which is activated $ZnCl_2$ at a contact time of 5 hours. This is caused by residual chemicals that are still attached to the carbon pores which were not completely opened during carbonization.

Table 1. Characteristics of ACHC with and without chemical activator

Activator	Activation Time (hour)	Moisture content (%)	Ash Content (%)	Adsorption Rate of Iod (mg/g)
$ZnCl_2$ 0,1N	1	4,90	6,40	888,37
	3	2,97	9,00	634,55
	5	6,93	14,89	571,09
$CaCl_2$ 0,1N	1	5,88	5,99	793,18
	3	4,85	8,45	666,27
	5	4,90	9,45	539,36
Non Activation (NA)		2,00	21,53	476,00
Indonesia Standard SNI 06-370-1995		15	10	750

3.3 Characteristics of Carbon Functional Group Using FTIR

The results of FTIR testing were carried out on 3 samples with the best characteristics, namely 0.1N ZnCl2 activated coffee skin carbon with an activation time of 1 hour; CaCl2 0.1N at 1 hour activation time; and FTIR testing after contact time with tofu liquid waste. The results of activated carbon surface analysis using FTIR analysis are shown respectively in Figures 2, 3 and 4.

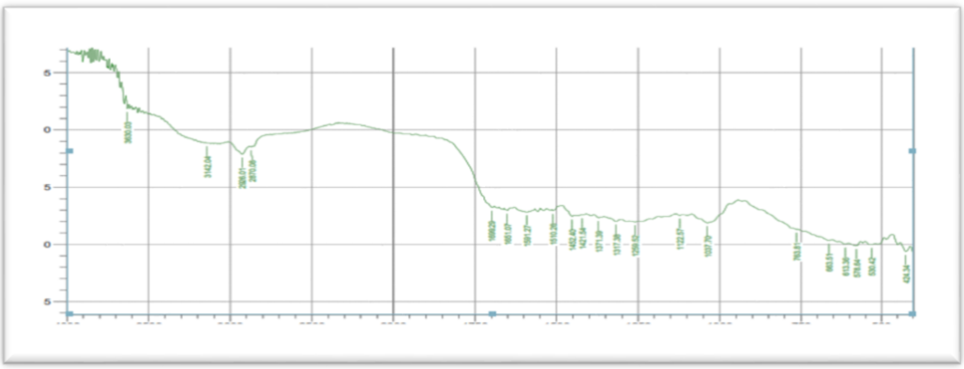

Figure 2. FTIR data of ACHC using activator $ZnCl_2$ 0,1N

Proceeding of ICMR 6(2), 279-289 DOI: https://doi.org/10.32672/picmr.v6i2.1273

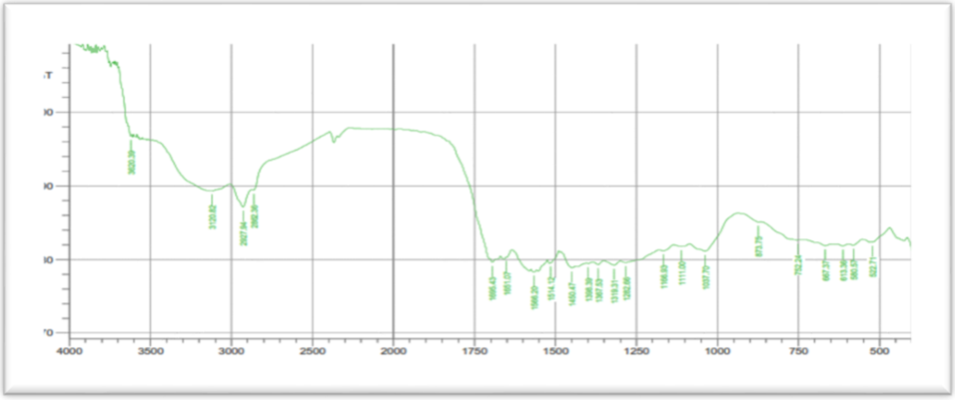

Figure 3. FTIR data of ACHC using activator $CaCl_2$ 0,1N

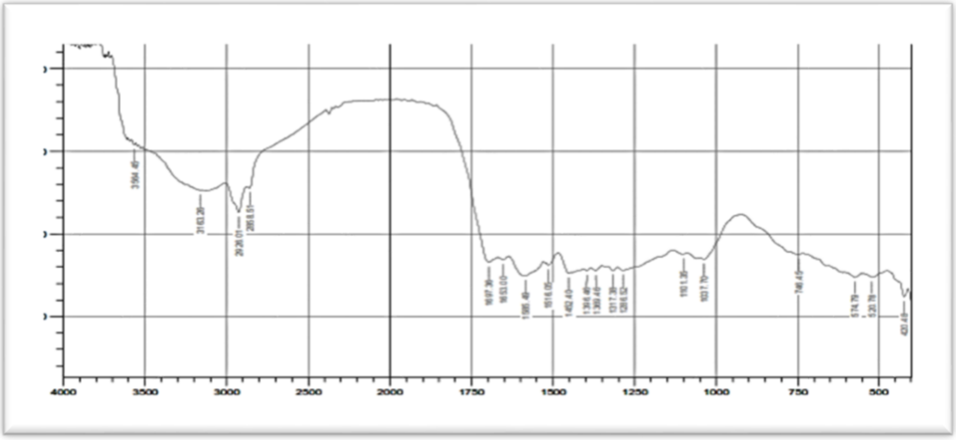

Figure 4. FTIR Data of activated carbon after contacted with tofu was water

At 1-hour activation time, wavenumber was identified at 2870-3564 nm and frequency of 85.559. The functional group of alkanes (C-H) were found at the surface of the coffee husk activated carbon. Whereas after contact time on tofu liquid waste, variations in alkane stretching (C-H) were found at wave numbers 2860-3620 with a frequency of 89.432. The activation highly affected the change of wavenumber found 2858-3564 and a frequency of 85,579, which is lower with a wavelength of carbon function before activation. In **Table 2**, the functional groups attached to the surface of ZnCl2 0.1N and CaCl2 0.1N chemically activated carbon were tabulated.

Table 2. Functional group of the highest peak found on the surface of arabica husk carbon

No	Sample	FTIR wavenumber range (nm)	Frequency	Functional Group
1	$ZnCl_2$ 0,1 N	2870 – 3564	85.559	Alkane (C-H)
		3142	70.516	Hidroxy (O-H)
2	$CaCl_2$ 0,1 N	2863– 3620	89.432	Alkane (C-H)
		3120	70,232	Hidroxy (O-H)
3	ACTWW	2858 – 3564	85.579	Alkane (C-H)
		3161	74.939	Hidroxy (O-H)

Note: ACTWW=Activated Carbon in Tofu Wastewater

Proceeding of ICMR 6(2), 279-289 DOI: https://doi.org/10.32672/picmr.v6i2.1273

The three FTIR results show similarities in functional groups, namely the presence of absorption in areas that indicate pore opening. In the three FTIR results produced, it shows that there is absorption in the area around 2870-3564, which indicates the presence of a double ring. However, the intensity is small, this indicates incomplete crystal growth (Sriatun, 2004). From the results of FTIR analysis we can conclude that the best activated carbon uses 0.1N ZnCl2 activator for an activation time of 1 hour. Meanwhile, compared to the 0.1N CaCl2 activator during the activation time of 5 hours and after contact time with tofu liquid waste, it was less good.

3.4 Effect of Adsorbent Contacted Time on the Tofu Wastewater Removal

3.4.1 pH Removal

The degree of acidity is a description of the amount or activity of hydrogen ions in water, one of the special characteristics of tofu liquid waste is that it has a pH value of < 7. A good pH level is a pH level that still allows biological life in the water to run well, a good pH for drinking water and wastewater is a neutral pH of 7 (Sugiharto, 2008). This pH value is important to consider because it can affect the process and speed of chemical reactions in water.

In activated carbon activated ZnCl2 0.1N, before being contacted activated carbon to the pH of tofu waste with a value of 3.8. After contact with tofu liquid waste for 1, 3 and 5 hours. Able to set aside pH from 3.8-4.4 at 1-hour contact time, at 2-hour contact time able to set aside pH value from 3.8-4.6 while at 5-hour contact time pH allowance from 3.8-4.7 is obtained.

The pH level in this study is still not in accordance with the quality standards of the Minister of Environment Regulation Number 5 of 2014 which states that the maximum level is 6-9. Roesiani's research (2015) states that the pH value of tofu liquid waste increases with variations in the length of contact with activated carbon, the study uses variations in the length of contact for 3 minutes, 5 minutes, and 7 minutes where the length of activated carbon obtained which is most effective in increasing the pH value of tofu wastewater is 7 minutes contacted time with the pH changes to 4.15. Comparing to this study, the pH level of tofu wastewater was increased to 4.7 on the length of contacted time for 5 hours. This shows that activated carbon can work effectively in increasing the pH value of tofu wastewater even though it has not met the quality standards of pH values of 6-9. In the use of activated carbon activated CaCl2 0.1N, an increase in pH was obtained from 3.8 before treatment to 4.5. At 1 hour contact time, at 3 hours contact time pH raised from 3.8-4.6, while at 5 hours contact time pH allowance from 3.8-4.7. From the research, the contact time with absorbent for 5 hours in tofu wastewater could lowering the pH from 4.5 to 4.7 at the activation time 5 hours.

3.4.2 COD Removal

COD is the amount of oxygen needed to oxidize organic substances in one liter of liquid sample with the oxygen source coming from chemicals. If a body of water has a large COD, this indicates that the water is polluted and contains organic substances, resulting in a reduction in the dissolved oxygen content in the water (Effendi, 2003). In Figure 5, the reduction in the COD value of tofu liquid waste was only carried out on activated carbon samples with the best absorption which were activated by $ZnCl_2$ 0.1N and $CaCl_2$ 0.1N for 3 hours.

Proceeding of ICMR 6(2), 279-289 DOI: https://doi.org/10.32672/picmr.v6i2.1273

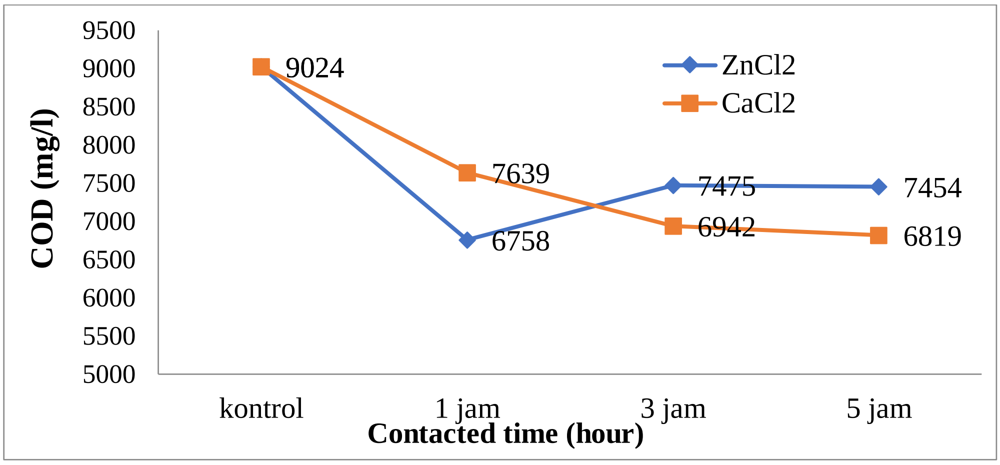

Figure 5. Effect of contacted time on the COD removal using ACHC activated with ZnCl2 and CaCl2

From Figure 5, carbon activated with 0.1N ZnCl2 activator is able to reduce COD in the range 9024 to 6758 or with an efficiency of 25% at a contact time of 1 hour. At a contact time of 3 hours it is able to reduce COD values in the range 9024 to 7475 mg/ l with a reduction efficiency of 17%, while at a contact time of 5 hours it was able to reduce COD levels with a range of 9024-7454 mg/l or with an efficiency of 17.4%. The best results from the 0.1N ZnCl2 carbon in reducing the COD of tofu wastewater were at a contact time of 1 hour, whereas at a contact time of 3 hours and 5 hours the COD level value increased, this is because if the contact time of the waste with activated carbon is extended Even though high pressure will not increase the number of adsorbed molecules, activated carbon that has absorbed waste optimally will release its adsorbate back into the blank or waste solution. Taib's research (2014), was able to reduce COD parameter of tofu wastewater by 982 mg/l with a percentage of 55% with a COD value before treatment was 2187 mg/l.

After being contacted for 1 hour, the COD removal levels using a 0.1N CaCl2 activator, the effectiveness in reducing COD in tofu liquid waste was in the range 9024-7639 mg/l or with an efficiency of 15%. At a contact time of 3 hours the level of carbon effectiveness could reduce the COD levels with a range of 9024-6942 mg/l or with an efficiency of 23%, while at a contact time of 5 hours, it is able to reduce COD values with a range of 9024-6819 or with a reduction efficiency of 24%. The most effective COD removal was at 5 hours of contact time. The longer the contact time, the better the absorption of liquid tofu waste.

In Figure 5, it can be seen that the COD of tofu liquid waste before undergoing treatment with activated carbon from coffee husk has a greater value than after treatment. Although the value has not decreased much and does not meet the Quality Standards for Industrial Waste Water No. 5 of 2014, the COD value before treatment was 9024 mg/l. The addition of activated carbon from coffee skins when contacted with tofu liquid waste using a variety of stirrers can reduce COD levels for 1 hour, 3 hours and 5 hours.

3.4.3 BOD Removal

BOD is a measure of the amount of organic substances that can be oxidized by aerobic bacteria or the amount of oxygen used to oxidize a number of organic substances in aerobic conditions (Darsono, 2017). The importance of the amount of oxygen in water makes it necessary to provide a measure of the oxygen requirements required by bacteria.

Proceeding of ICMR 6(2), 279-289 DOI: https://doi.org/10.32672/picmr.v6i2.1273

One measure is particle size in the form of Biological Oxygen Deman (BOD, oxygen demand for biological needs). Figure 6 shows a graph of the relationship between contact time and the reduction in BOD of tofu liquid waste activated with 0.1N $ZnCl_2$.

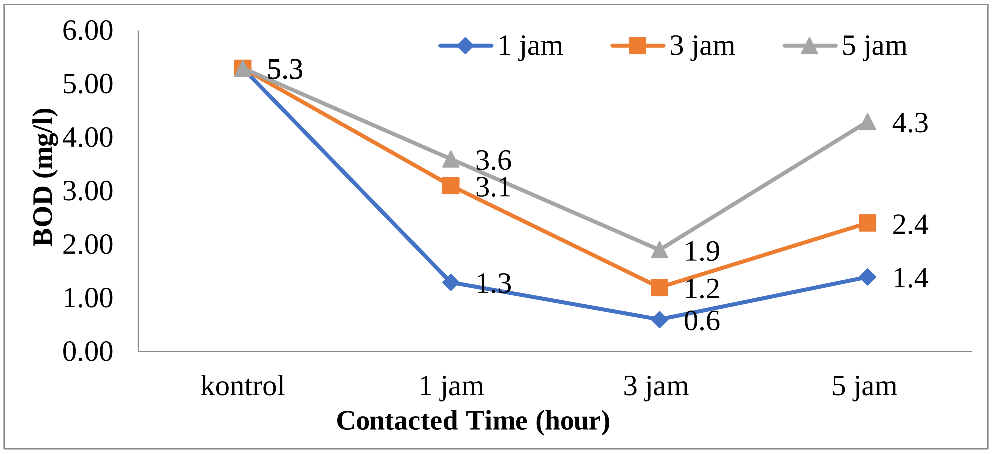

Figure 6. Effect of contacted time and the reduction in BOD using carbon impregnated with 0.1N $ZnCl_2$

In Figure 6, it showed the BOD value before treatment was with a BOD value of 5.3, after treatment, it was able to reduce BOD by 5.3-1.3 mg/l or with an efficiency of 75% at a contact time of 1 hour, at a contact time of 3 hours. able to reduce BOD from 5.3-0.6 mg/l or with an efficiency of 88%, while at a contact time of 5 hours from 5.3-1.4 with an efficiency of 73%. From the contact time value, we can conclude that the best BOD value is coffee skin activated carbon with a contact time of 3 hours with tofu liquid waste. The BOD value increased at a contact time of 5 hours, this shows that the activation time of the coffee skin activated carbon is less effective because fewer activated carbon pores are formed to absorb organic substances. Reducing organic substances in tofu liquid waste will reduce BOD because the oxygen used by microorganisms to decompose these organic substances for 5 days is reduced.

In Figure 7, a graph shows the relationship between contact time and the reduction in COD of tofu wastewater using the carbon impregnated with 0.1N CaCl2.

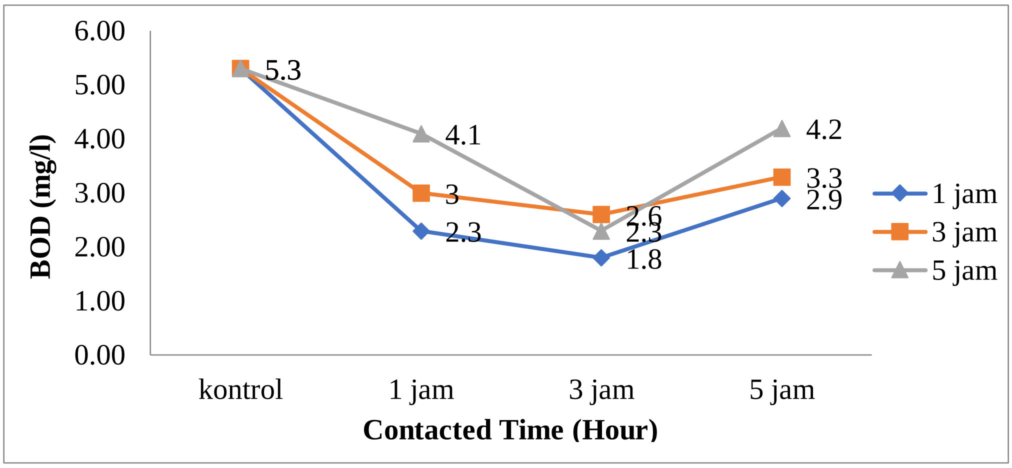

Figure 7. Effect of contact time on the BOD removal using carbon activated with $CaCl_2$ 0,1 N

Proceeding of ICMR 6(2), 279-289 DOI: https://doi.org/10.32672/picmr.v6i2.1273

From Figure 7. it showed the BOD of tofu wastewater before treatment with activated carbon from coffee husk has a greater value than after treatment with activated carbon at 5.3 mg/l. After activated carbon treatment it was able to reduce the BOD of tofu wastewater from 5.3-2.3 mg/l at a contact time of 1 hour or with an efficiency of 56%, at a contact time of 3 hours from 5.3-1.8 mg/l with an efficiency 66%, while at a contact time of 5 hours activated carbon was able to reduce BOD from 5.3-2.9 mg/l or with an efficiency of 45%. We can conclude that the BOD value is best for absorbing BOD from tofu liquid waste at a contact time of 3 hours. According to Handayani (2005), this decrease was caused by the rate of formation of adsorbent and adsorbate complexes being proportional to the rate of decomposition of adsorbents and adsorbate into molecules. So, after maximum adsorption occurs, decomposition will occur. Meanwhile, at a contact time of 5 hours, the BOD value increases, this is because the longer the contact time, the more saturated the active carbon becomes in absorbing organic contaminants from tofu wastewater.

Based on the research results above, it can be concluded that the type of activator and variations in contact time greatly influence the characteristics of the coffee skin activated carbon produced and the contact time influences the reduction in pH, BOD and COD in tofu liquid waste. So it is recommended that in future research, the activated carbon activation time be taken using a different activator with a time variation of less than 1 hour and it is recommended that in future research carry out an analysis with a variation of contact time of less than 5 hours.

4. Conclusions

The results of the study concluded that coffee husk waste has the potential to be used as a raw material for making activated carbon. The characteristics of coffee skin activated carbon before and after chemical activation including moisture content, ash content and iodine uptake have met SNI standards (06-370-1995). FTIR test before and after contact time with tofu waste did not have a significant effect on changes in activated carbon functional groups. The contact time of the coffee skin adsorbent, namely 1, 3 and 5 hours, was able to increase the pH by 3.8 to 4.7, reduce the BOD content by 88%, and COD by 25% for 0.1N $ZnCl_2$ activated carbon. Meanwhile, 0.1N $CaCl_2$ activated carbon was able to increase the pH from 3.8 to 4.7, reduce BOD by 66%, and COD by 24%.

To improve the adsorption capacity of COD content of tofu wastewater more effectively, it is necessary to carry out further research using a variety of chemical activators with higher concentrations and varied masses of activated carbon. It is hoped that the activated carbon product from Gayo coffee husks produced can be applied later in the water purification process and the absorption of others industrial wastewater in Indonesia.

5. Acknowledgments

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