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Use of Microwave Radiation for Activating Carbon from Rice Husk Using $ZnCl_2$ Activator

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Abstract

Activated carbon is one of the most commonly used adsorbents in a variety of separation processes because it is inexpensive, and also the design and principal of application are quite simple. The ability of activated carbon as an adsorbent is related to its large surface area and pore volume, varying pore structure, and diverse surface reactivity. The use of microwave radiation can further improve the efficiency of activated carbon adsorption. Micro-waves can affect the pore texture and surface of the activated carbon, but rarely do both practitioners and researchers control these variables influencing the relationship between features and performance of biomass-based activated carbon as an adsorbent at the time of manufacture from the initial stage (carbonation) to carbon application active (e.g., adsorption of heavy metals, surfactants, and organic molecules). This study aims to synthesize activated carbon from rice husk, which has the efficiency and capacity of adsorption of heavy metals such as Pb and activator organic molecules used is $ZnCl_2$ 30% and microwave radiation. This research has succeeded in making activated carbon using the $ZnCl_2$ activator and microwave radiation. The time and power of microwave radiation that provides the highest efficiency in the carbon activation process for Pb ion adsorbate, were 7 minutes and 800 W. For phenol adsorbate was 5 minutes at 800 W. The highest efficiency time and concentration of adsorption for Pb ion adsorbate were 40 minutes at 60 ppm while for phenol adsorbate were 5 hours at 100 ppm. The adsorption efficiency for Pb cation adsorbate was 99.57%. While for phenol adsorbate is 81.05%. Characterization with FTIR, SEM-EDX, and SAA showed a C-Cl bond, the pores were visible, and an increased surface area of activated carbon was 36.9 times the surface area of carbon and the pores formed were mesoporous.

1. Introduction

Rice husk is a material that contains a high level of carbon. The components contained in rice husks include 39.8-41.1% carbon, 5.7-6.1% hydrogen, 0.5-0.6% oxygen, and 37.4% -36.6% nitrogen. The high carbon content in rice husks is because rice husks are lignocellulose precursors that contain lots of cellulose, hemicellulose, and lignin [1].

Activated carbon is an anamorphic carbon compound, which can be produced from materials containing a high level of carbon or from charcoal and treated in a distinctive way to get a wider surface area. The surface area of activated carbon ranges between 300-3500 m^2/gram, and this is related to the internal pore structure that causes activated carbon to have an adsorbent property. Activated carbon can adsorb certain gases and chemical compounds or a selective adsorption property, depending on the size or volume of the pores, and also surface area.

$ZnCl_2$ is an activator that functions as Lewis acid, which could bind compounds such as alkaline and also

inhibit the formation of volatile compounds, thereby increasing the acquisition of activated carbon [2]. That causes a more significant acquisition of activated carbon in the synthesis using $ZnCl_2$. The resulting activated carbon pore depended on the ratio of $ZnCl_2$ that were combined during activation. The concentration ratio of $ZnCl_2$: Biomass 1: 5 concentration ratio, most active carbon pores classified as microspore structures (<2 nm), while at 2: 5 and 3: 5 ratios, the structures become mesoporous (2-50 nm) [3].

The activation between $ZnCl_2$ and carbon was carried out in this research using microwaves. Activation of carbon using microwave irradiation has several advantages, which has a high efficiency of power consumption, varying heating rates in a relatively short period of heating times so that energy consumption becomes lower. Most important is that micro-wave effected to the pore texture, and also the surface of activated carbon [4]. The activated carbon obtained was applied to absorbed heavy metal ions (Pb) and phenols since these two adsorbents are pollutants.

This research aims to determine the time and power of micro-waves in the carbon activation process, determining the optimal conditions of the activated carbon during the adsorption process of heavy metal ions and phenols, and also determining the efficiency and capacity of activated carbon adsorption for these two adsorbate ions.

2. Methodology

2.1. Material and Equipment

The equipment that is needed in this research is a standard set of laboratory glassware, Pyrolysis reactor, AT 200 *Mettler* scale, 630F Isotope Oven, UV-Vis Shimadzu UV-1201 spectrophotometer, 100 mesh sieves, microwave, Surface Area Analyzer (SAA) and Atomic Absorption Spectrophotometry (AAS). The materials used in this research are rice husk, $Pb(NO_3)_2$, phenols, distilled water, $ZnCl_2$ solids.

2.2. Research Procedure

Carbon is obtained from the carbonization of rice husks by pyrolysis at a temperature of 300°C for 10 minutes. The carbon obtained was then put into 100 mL 30% $ZnCl_2$, and irradiated using microwaves with various set of time starting from 1, 3, 5, 7, and 9 minutes, with the power of the microwave ranging from 80, 240, 400, 640, to 800 W. Activation time and radiation power of the carbon activation process was determined as a preliminary test in order to obtain the optimum radiation time and power used for the adsorption process.

Activated carbon is then washed with distilled water until it reached a constant pH level, then dried at a temperature of 105°C for 10 minutes, after the drying process it is crushed then sieved with a 100-mesh sieve, then it was contacted with the adsorbate.

Pb ions with varying concentrations of 60, 75, 100, 125, 150 and 175 ppm, and phenols with variation in concentrations of 50, 100, 200, 300, 400, 500 ppm contacted with activated carbon with a range of contact time ranging between 20-60 minutes for Pb ions, and 1-6 hours for phenol. Pb and phenol metal ions that were absorbed were each determined by AAS and UV-Spectrophotometer.

Activated carbon before and after adsorption were characterized using FTIR, surface area analyzer (SAA), and SEM-EDX.

3. Result and Discussion

Microwaves are a new alternative way to produce heat by converting electromagnetic energy into heat, compare to the conventional conductive heating processes. That indicates that liquids, solids, and chemical reactions can be caused by microwave dielectric heating. Microwave dielectric heating caused by the polarization of dipolar molecules. The dipolar polarization mechanism created by the dipole moment in the material, when there is an external electric field, this dipole moment will adjust to having the same direction as the electric field, then when there is microwave radiation, the electric field will reverse direction, so that the dipole will oscillate and produce external dielectric energy, then this energy will be removed by friction so as to produce thermal energy [5].

The carbon activation process uses $ZnCl_2$ activator carried out under microwave radiation that will produce activated carbon that has more active sites, in a shorter time, able to open pores that were not accessible before, creating new pores with selective activation and widening and merging of the pores through the damaged of the pore wall. The change in porosity will result in high activation efficiency [6].

3.1. Activated Carbon Procedure

This research started with the carbonization process; in general, the carbonization process causes organic compounds present in rice husk to undergo the pyrolysis process so that carbon is formed, which also causes a significant change to the weight of the material due to decomposition of organic compounds. The volatile pyrolysis compounds that were produced during the process will be released, carbon and other organic compounds will be left as carbon [7].

During the pyrolysis process, the material will undergo decomposition, the chemical bonds in the material break off thermally, and volatile compounds will be produced during the process. The volatile compound is the result of the volatilization process, usually consisting of three types, namely: gas (CO, H_2, CO_2, H_2O, and CH_4), tar (CH_3OH, CH_3COOH, benzene derivatives) and charcoal (stable carbon). The three types of volatile compounds produced by pyrolysis or devolatilization depend on the composition of the material and operating conditions.

The pyrolysis process consists of several stages, first is the evaporation of water at 100-200°C, then the breakdown of macromolecular structures into gas and solid carbon at 200-380°C, and lastly the depolymerization and breakdown of C-O and C-H chains, and also the conversion of organic liquid components into tar at 380-400°C [8]. The process of pyrolysis takes place slowly and will form large quantities of carbon. The series of physical and chemical reactions occurred gradually at temperatures below 350°C, and this process is the absorbing of heat (endothermic)[9].

The activation process from the carbonization of rice husk is carried out through chemical activation using $ZnCl_2$ 30%, and physical activation is done by using microwave radiation with the time and power of radiation being varied. Variation of time and power was done to determine the optimum radiation time and power. The function of the activation process was so that the activated carbon produced has large pores and a high capacity of absorption. High porosity will increase the absorption capacity of activated carbon.

Activated carbon that uses $ZnCl_2$ as an activator can produce carbon with micro and mesoporous pore size [10]. Activation using $ZnCl_2$ causes swelling in the molecular structure of cellulose. The swelling of the structure causes the lateral bonds in cellulose to break, thereby increasing the cavity in the carbon produced [11].

Activation via microwave heating can be explained as dipolar polarization and ionic conduction. Most solvents have a permanent dipole. When microwaves are applied, and there is a change in the electric field, the dipole rotates, which causes friction and heat. The ions in a solution can also produce heat by ionic conduction [12]. The carbon material is a good absorber of microwaves and has a high capacity to absorb and convert microwave energy into heat. Microwave energy is very efficient at heating a material because there is no power wasted [13]. Activation via microwaves also requires a short amount of time compared to conventional methods due to the fast process and the evenly generated heat [14]. On activation with $ZnCl_2$ through microwave radiation, heat given by microwaves causes carbon to open so that Cl can bond with carbon to form C-Cl bonds. $ZnCl_2$ activator encourages dehydration; in addition, $ZnCl_2$ also acts as a conductor of heat by absorbing microwaves in the initial stages of radiation in the activation stage [15]. After the activation process, carbon washing is needed to remove any residual organic residues, contaminants, minerals, and metal remnants left in the pore cavity. Drying the results of carbon activation at a temperature of 105°C for one hour aims to help the release of H_2O, CO, CO_2, and CH_4, which is the result of the activation and carbonization process [16].

3.2. Effect of Microwave Radiation Time on Adsorption Efficiency

Determining the optimum time for microwave radiation activation is done by contacting activated carbon with Pb ions and phenols. Determining the optimum radiation activation time is done by looking at the ability of activated carbon to adsorb Pb ions at a concentration of 70 ppm and phenol at a concentration of 300 ppm. The results of the analysis, the adsorption of activated carbon with variation in microwave radiation time, and fixed power of 400 Watt to Pb ions and phenol shown in Figure 1.

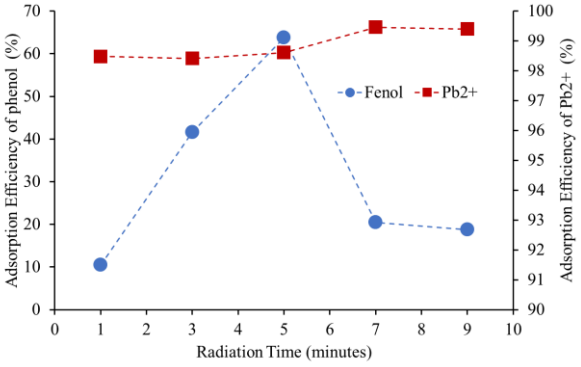

Figure 1. Interaction between activation time with adsorption efficiency of Pb ion and phenol.

The time of activation influences the development of the pore structure. There are no significant changes in the adsorption efficiency of Pb ions by activated carbon that were exposed from 1 to 9 minutes of radiation. In the initial stages, both microspore and mesoporous increase significantly, which indicates that the pores are formed and enlarged simultaneously [17]. The longer radiation time is applied, it causes an increase in the rate of reaction and the development of the undeveloped pore structure [18]. The percentage increase in adsorption efficiency with the exposer of 1 to 9 minutes is only 0.92%. That is because in the 1-minute radiation, an active site has been formed on the surface, and the pores in the activated carbon are suitable for Pb ion adsorption [6]. The tendency of Pb ions adsorption efficiency is constant due to the pores formed are not significant, so the adsorption that occurs does not show any significant differences [17]. Even so, at an exposure of 7 minutes showed the highest efficiency, amounting to 99.46%.

Figure 1 also shows that the optimum radiation time for phenol is 5 minutes because the highest peak obtained at 5 minutes with an adsorption efficiency of 63.77%. At 5 minutes, there is an opening of new pores that were previously inaccessible and the formation of new pores [6, 18]. Then in the 7 minutes' mark, the efficiency drops because it passes the optimum time for heating, and this destroys pores in the surface of activated carbon [19]. The problem between the adsorption of Pb ions and phenols in the radiation time needed to reach a certain percentage, the radiation time of Pb ions for 1 minute, can achieve 98.48% adsorption efficiency while for phenol at 5 minutes the adsorption efficiency is only 63.77%. Pb ion is derived from $PbNO_3$, whose solubility in water is 52 g/100 mL (20°C), but phenol has limited solubility in water, which is 8.3 grams/100 ml. It shows that there is an influence of polarity of the adsorbate with the time of activation affecting the surface properties of activated carbon, impurities on the surface are released so that the active site is more open, which causes the surface of

activated carbon to be slightly polar. The second reason is that the radius of Pb ion is much smaller than the phenol. The Pb ion was adsorbed inside the microspore and the mesoporous that is formed by radiation, while phenol is more adsorbed on the outer surface or in pores that are the size of phenol diameter.

3.3. The Effect of Microwave Radiation Power to Adsorption Efficiency

Determining the optimum activation radiation power is done by looking at the ability of activated carbon to adsorb Pb and phenol ions, it can be seen in figure 2. With the increase of microwave power output, it resulted in an increase in the percentage of Pb and phenol ion adsorption efficiency. The highest efficiency of adsorption of Pb ions and phenol occurs at 800-Watt power, reaching up to 83.34% for Pb ions and up to 87.35% for phenol adsorption. The higher radiation power will increase the adsorption efficiency due to the development of the pore structure, and this shows that the radiation power plays an essential role at the activation stage [6]. Optimum power can also show the heat generated in the process of making activated carbon, which is equal to 800 J/s or 0.191 kcal/s.

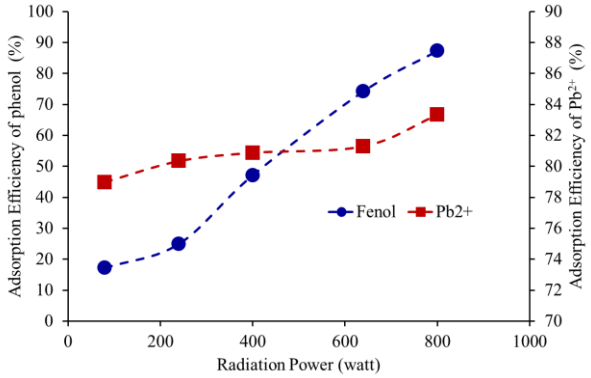

Figure 2. The relationship between radiation power and the adsorption efficiency of Pb ions and phenols.

3.4. The Effect of Adsorption Contact Time on Adsorption Efficiency

Contacting 1 gram of activated carbon at the contact time of 20, 30, 40, 50, and 60 minutes showed that the highest adsorption efficiency occurred in the 40 minutes' mark with a percentage of adsorption efficiency reaching 94.80%. The effect of adsorption contact time on the adsorption efficiency of Pb ion by activated carbon shown in Figure 3.

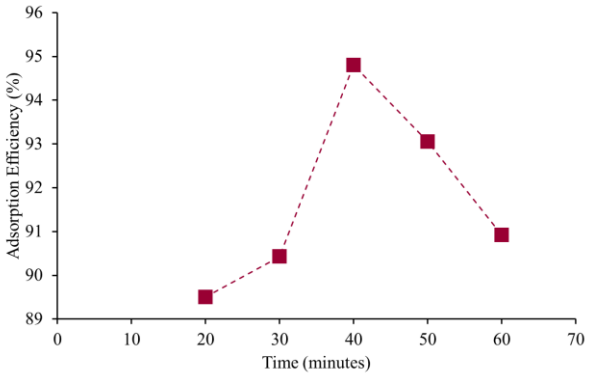

Figure 3. The effect of contact time on Pb ion absorption.

Based on the diagram in figure 3, the effect of contact time on Pb ion absorption shows that the longer the contact time, the higher the adsorption efficiency, the highest percentage occurs at 40 minutes' contact time. Pb ions enter the adsorbent cavity in the adsorption process, the longer the contact time, the more Pb ions fill the surface and pores of the adsorbent. At the contact time of 50 and 60 minutes, there was a decrease in the adsorption efficiency. That could be caused that activated carbon has begun to saturate, increasing contact time decreases the ability of activated carbon in absorbing Pb ions because the pores and surfaces of activated carbon are filled or Pb ions are depleted.

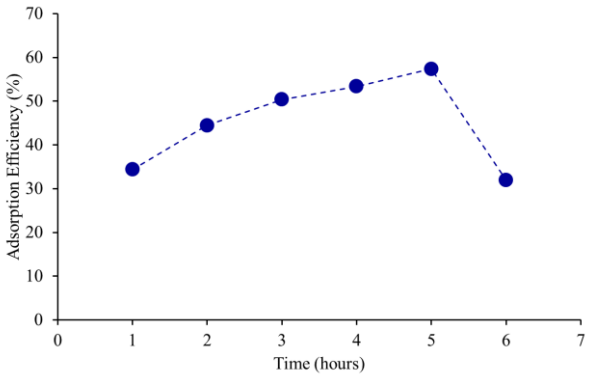

Figure 4. The relationship between adsorption contact time and phenol adsorption efficiency.

Figure 4 shows that the longer contact time will increase the efficiency of phenol adsorption; this is related to the increasing number of phenols that interact with the active site on the adsorbent surface. At 5 hours, the phenol adsorption reaches its optimum point; this is the following research that's been conducted [20]. In contrast to the Pb ions, the contact time of adsorption is shorter than the contact time of adsorption of phenols; this is because phenols have larger molecules than Pb ions, so that it is more challenging to adsorb than Pb ions which can directly enter the pores of activated carbon.

3.5. The Effect of Adsorbate Concentration on Adsorption Efficiency

Contacting 1 gram of activated carbon with varying concentrations of 60, 75, 100, 125, 150, 175 ppm shows that the highest efficiency percentage is 99.57% for Pb

ion. The concentration is 60 ppm. The effect of concentration can be seen in Figure 5.

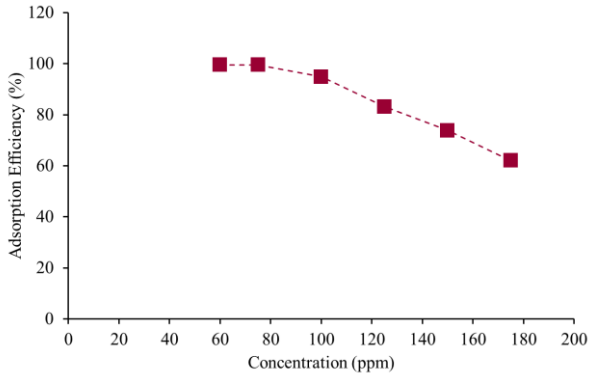

Figure 5. Relationship of initial Pb ion concentration with adsorption efficiency.

Based on Figure 5, it can be seen that the higher the concentration, the percentage of adsorption efficiency decreases. At low concentrations, the Pb ion in the solution will interact with the active site so that the absorption occurring is almost 100%. Increased concentration causes more Pb ions not being absorbed by activated carbon due to saturation at the active site [21]. Contact was also tested on carbon without activation to Pb metal ions at a concentration of 60 ppm with an optimum time of 40 minutes, with an adsorption efficiency of 10.71%. The effectiveness of inactivated carbon adsorption is smaller than that of activated carbon. That indicates that the activation process that was done is successful in increasing the capacity of carbon to adsorb Pb ions, which increases its efficiency

Some possible mechanisms that can occur in the adsorption of Pb ions by activated carbon includes ion exchange with metal cations, H^+ from hydroxyl acid and carboxyl of activated carbon exchanged with Pb ions, the -OH or C=O functional groups provide active sites for complexation surface with Pb, mesoporosity of activated carbon which accelerates the diffusion of Pb ions [22, 23]. The activated carbon pores play an essential role in the adsorption process, through the SAA characterization (Table 2) it is known that the radius of activated carbon is 16.259 Å. The size of the Pb ion radius is 1.8 Å so that the pore radius produced by activated carbon allows the Pb ion to enter the pores. In addition, it is possible that the C-Cl bond formed by the use of $ZnCl_2$ can also play a role in the adsorption process in which the Cl group can bind to Pb ions [24].

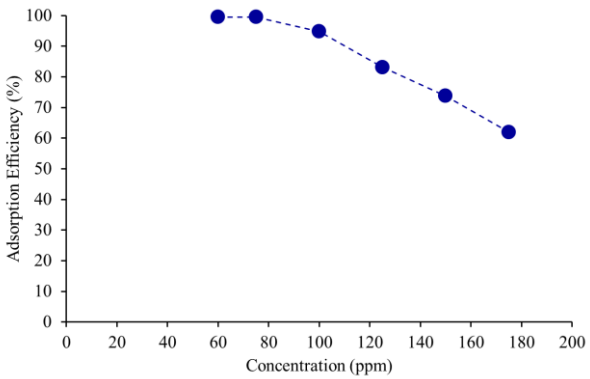

Figure 6. A graph of the relationship between initial phenol concentrations and adsorption efficiency.

Figure 6 shows that an increase in the efficiency of phenol adsorption is occurring at a concentration of 50 to 100 ppm; this is due to increased resistance to overcome the difficulties between phenol mass transfer to the adsorbent, thus that it will increase the interaction between phenol and also activated carbon. The decrease in adsorption efficiency when the concentration is increased to 200, 300, 400, and 500 ppm, this shows that 100 ppm is the optimum concentration of phenol adsorption using activated carbon. This decrease inefficiency is due to the active site on the surface of activated carbon being saturated so that it can no longer react with phenol, besides the adsorbed phenol also undergoes desorption due to unstable interactions [25].

3.6. Characterization with FTIR Spectroscope

Characterization using FTIR spectroscopy aims to determine the functional groups that are inactivated carbon as a comparison of carbon and activated carbon that adsorbed Pb ions, and both are characterized. Based on Figure 7, it shows the spectra of carbon, activated carbon, and activated carbon that have been contacted with Pb ions have almost identical peaks. After fitting using fityk at wavelengths of 700-1400 cm^{-1}, the result shows that there was a difference in the peaks that indicate the existence of certain groups. The difference is the existence of a peak at a wavelength of 832.6 cm^{-1}, which suggests the presence of C-Cl bonds after the activation process using $ZnCl_2$.

Pictures of carbon activated carbon, and activated carbon that has been contacted with Pb ions can be seen in Figure 8. There are some differences in the infrared spectra of carbon that have absorbed Pb ions compared to activated carbon, which is the shift and decrease in peaks at a wavelength of 1103.28 and 1604.77 cm^{-1}, which showed the influence of CO and C=O groups in the adsorption of Pb ions.

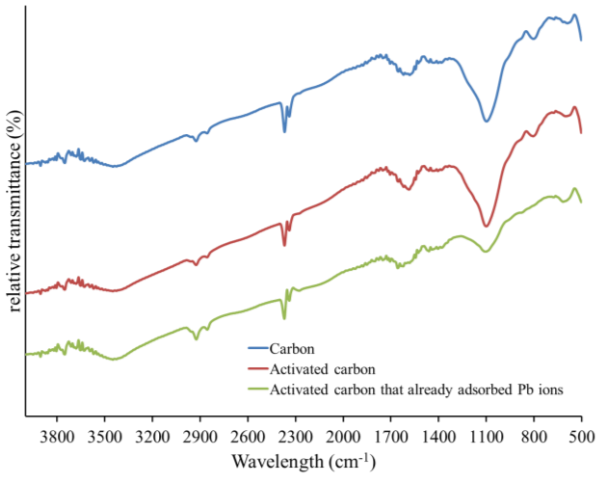

Figure 7. FTIR carbon spectra of activated carbon and activated carbon which has absorbed Pb ions

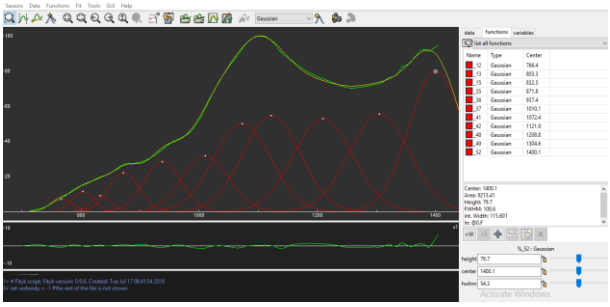

Figure 8. The Result of Fityk spectra FTIR on activated carbon which has absorbed Pb ions

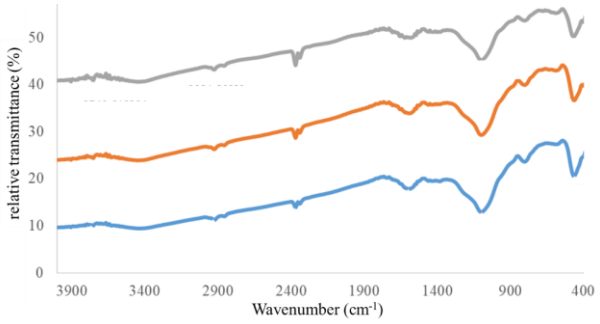

Figure 9. FTIR spectra on Carbon, Activated Carbon, and Activated Carbon-phenols.

Figure 9 shows that there are similarities between the FTIR spectra of carbon, activated carbon, and activated carbon that adsorbed phenols. That can be seen in the presence of peaks at 802.39 cm^{-1}, 1581.62 cm^{-1}, 1095.57 cm^{-1}, and 3427.57 cm^{-1}, which indicates the presence of Si-H, Si-O, CO, C = C aromatic and –OH. In carbon before activation was found a peak in wavelength number of 1851.66 cm^{-1} which indicates the presence of anhydrous carboxylic groups due to imperfect carbonization process, then this peak disappears after activation, this shows that the activation process results in the decrease in the number of functional groups on the surface of activated carbon so that aromatic ring size will increase and will increase carbon porosity. Whereas in activated carbon that has been used to absorbed phenol, a change in the wavelength number can be seen after deconvolution utilizing the application of the wavelength of 3626.17 cm^{-1} to 3632.27 cm^{-1} and at a wavelength of 3680.18 cm^{-1} to 3653.12 cm^{-1} indicating the presence of secondary -OH to the presence of phenol. That is consistent with research conducted by Ingole and Lataye [26]. Phenol adsorption can also be identified by changing wavelength numbers around 1090 cm^{-1} and 800 cm^{-1}. After being used for phenol adsorption, the peak shows Si-OH bonds shifted from 805.36 cm^{-1} to 814.79 cm^{-1}. In addition, there was a group that showed the Si-O-Si bond changed from the original 1094.35 cm^{-1} to 1087.67 cm^{-1}.

3.7. Characterization with SEM-EDX

Characterization using SEM-EDX was carried out at magnification of 10000 times. Figure 10a shows that there are pores on the surface of activated carbon due to chemical activation. These pores are formed due to the presence of $ZnCl_2$, which evaporates, leaving a cavity or pores, displacing the space of $ZnCl_2$. SEM characterization was carried out combined with EDX to see the pores and analyze the elements contained in the sample so that the effect of adsorption can be found in the composition of the elements in the sample. Figure 10 shows that in the visible pores open before adsorption (Figure 10a) and after adsorption (Figure 10b and c), the activated carbon pores have been covered by adsorbates (Pb ions and phenols).

Table 1. Composition Analysis of activated carbon samples

Elements	Weight percentage (%)	
	Before Adsorption	After Adsorption
Si	6.7	10.9
C	68.1	73.8
O	22.8	14.1
Cl	0.5	0.3
Zn	1.9	0.8
Pb	-	1.0

The results of the analysis using EDX is shown in Table 1, and the activated carbon contained 0.5% of Cl and 1.9% Zn by weight of the activated carbon. The data shows that the activated carbon samples still contain Zn and Cl elements, as well as activated carbon after adsorption, this proves that a successful carbon activation is characterized by the presence of Zn and Cl elements in the EDX spectrogram, this is consistent with research conducted by Allwar *et al.* [27]. In addition, it can also be seen that the composition of element C changes after adsorption. Element C increases from 68.1% before adsorption to 72.3% after adsorption, and this is due because phenol has been adsorbed on the surface of activated carbon so that it will change the weight percentage of element C. On activated carbon that adsorbed Pb ions also shows the Pb of 1.0%.

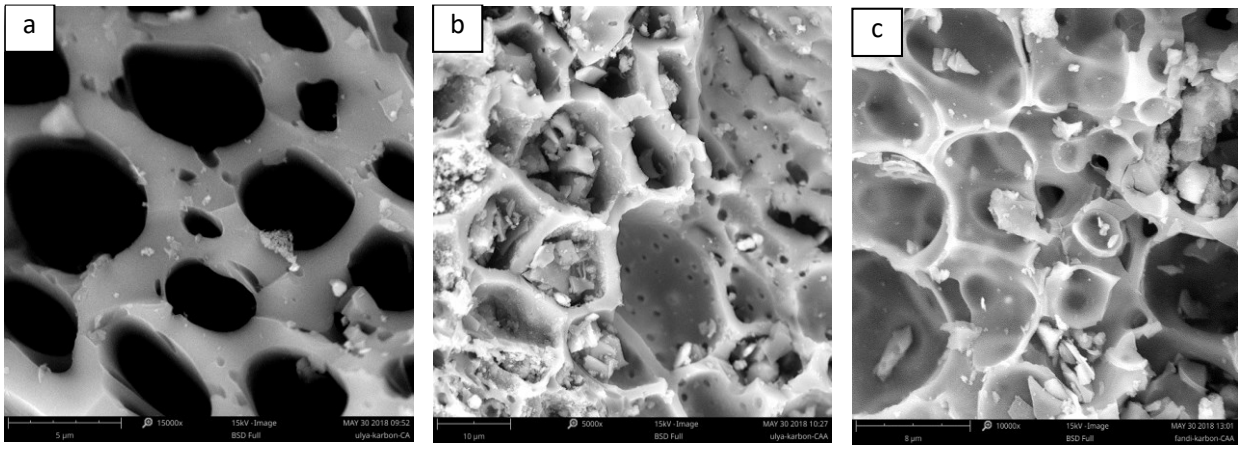

Figure 10. SEM-EDX a. Activated carbon, b. Activated Carbon of Pb ion, c. Activated Carbon of Phenol.

3.8. Characterization with *Surface Area Analyzer* (SAA)

The SAA analysis technique uses the Brunauer-Emmett-Teller (BET) method to determine the surface area and pore volume of activated carbon. The results of the analysis using BET can be seen in Table 2.

Table 2. Comparison of surface area and pore volume of activated carbon and carbon

Samples	Surface area (m^2/g)	Pore Volume (cc/g)	Pore Radius (Å)
Carbon	1.937	0.095	15.153
Activated Carbon	73.208	0.108	16.259

The results of the SAA analysis (Table 2) show that the surface area of activated carbon was 73.208 m^2/gram, and the pore volume was 0.108 cm^3/gram. While carbon without activation has a surface area of 1,937 m^2/gram and a pore volume of 0.095 cm^3/gram [28], that shows that activation using $ZnCl_2$ was able to increase the surface area by 36.9 times and the pore volume and pore radius also increased by 11.36 times and 1.07 times respectively. The results of the SAA analysis also show that the pore has an average diameter of 33,885 Å and also pore size distribution on the surface of activated carbon. The results obtained are shown in Figure 11. The pore distribution graph shows the most pore distribution has a diameter below 100 Å, and this indicates that the pore formed mesoporous size in the range of 50-200 Å [10]. Compared with a Pb ion diameter of 3.5 Å, the activated carbon pores are much larger so that the Pb ion can enter or be absorbed in the pores and the phenol size is larger than the Pb ion, and it is estimated that the phenol is absorbed only on the surface outside the activated carbon.

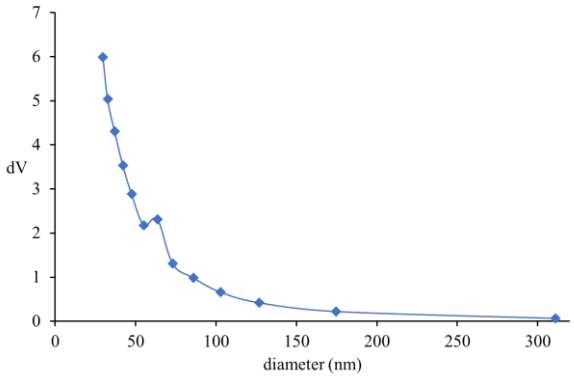

Figure 11. Graph of active carbon pore distribution

4. Conclusion

Microwaves and $ZnCl_2$ can be used to make activated carbon. The microwave radiation time and power which provides the highest adsorption efficiency in the carbon activation process for Pb cation adsorbate were 7 minutes at 800 W. While for phenol adsorbates was 5 minutes at 800 W. The highest concentration of adsorption with the highest adsorption efficiency for Pb ions was 40 minutes at 60 ppm while for phenol adsorbates were 5 hours at 100 ppm. The adsorption efficiency for Pb cation adsorbate was 99.57%. While for phenol, adsorbate was 81.05%. Characterization with FTIR, SEM-EDX, and SAA showed a C-Cl bond, the pores were visible, and an increased surface area of activated carbon was 36.9 times the surface area of carbon and the pores formed were mesopores.

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