Acid Mine Drainage Treatment Using Activated Carbon Ceramic Adsorbent in Adsorption Column

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Abstract——**Using powdered activated carbon as the adsorbent is very common nowadays, but the problem arises due to its light structure makes it easily washed out after or during the adsorption process. This research provides a method to combine powdered activated carbon with clay and modify them into ceramic adsorbent, the combination is molded into small balls and then baked in a furnace at a high temperature of 1000 °C. Activated carbon has been known for so many years as an effective adsorbent, but its use to adsorb heavy metals in acid mine drainage needs improvements to meet our expectation, chemical activation is one of the methods should be applied to enhance its adsorption capacity. The adsorbent is activated using alkali solution of NaOH 48% to fill its negative charge to enhance its ability in capturing heavy metallic cations contained in acid mine drainage. This research has proved that the adsorbent used is highly effective to reduce heavy metals pollutant in acid mine drainage, its adsorption capacity reaches most favorable results of 72.33% for Mn and 98.81% for Al at activated carbon:clay ratio 11:29 on contact time 300 min where pH increase is from 4 to 7.1. This may prove that the activated carbon ceramics adsorbent is effective enough to solve the acid mine drainage problem.**

Keywords— **acid mine drainage; heavy metal; activated carbon; clay; ceramic adsorbent; adsorption**

I. INTRODUCTION

Materials with a highly toxic level such as heavy metals were investigated in some of the sedimentations in the river [1], agricultural soil [2] and acidic leachate [3]. The cause of contaminations has generally been by mining activities [2] and landfills [4]. A number of studies have been conducted for toxic heavy metals removal from aqueous solutions. Controlling the pH of the solution is used in precipitation [5], ion exchange mechanism [6,7], and electrocoagulation [8], these are widely used to reduce the contamination level. Another attractive option is using adsorption method for heavy metal removal.

Accumulation of acid mine drainage containing heavy metals is going to give a bad impact to the environment especially those who live nearby mining area. Emerging health issues due to acid mine drainage should be controlled or diminished. There are many possible ways to solve this problem, one of them is by using adsorption method which is considered quite effective and relatively inexpensive.

The functional environmental for clean technology has received a growing interest, there have been researches actively on materials for air pollution control, air purification and wastewater treatment [9], [10]. There are numerous options but activated carbons are commonly used in environmental sector regarding the outstanding gas and liquid adsorption capacity and their supportive pore characteristics [9]-[12]. Activated carbons are prepared from both physical or chemical activation mechanism [13], [14]-[19]. Physical activation conducted through oxidation with hydrocarbon gas [16], vapor [17], carbon dioxide [18], or oxygen (air) [20] at high temperatures. The chemical activation forms activated carbons through dehydration method and oxidation reactions induced by the activators [14], [19], chemical activation can be conducted at relatively lower temperatures.

A simple carbonization method is performed to develop porous carbon. The temperature range for carrying the carbonization is 400-800 °C. Porous carbons are usually prepared from raw carbonaceous materials with two steps: carbonization and activation. In the carbonization process, pyrolysis releases most of the non-carbon components of the raw materials but leaving a rigid carbon skeleton with fragile textural properties. After the carbonization process, the activation procedure is performed using additional agents, they are: steam, CO_2, air, KOH, and $ZnCl_2$ at high temperatures to create the desired porous surface area [10]-[23] It requires a lot of effort and time in the activation step to determine the porosity of the carbon material.

Activated carbons advantages are large surface and porous areas existence with great volumes to adsorb high

amount of pollutants. Activated carbons can be made from waste materials, therefore, they are considered low-cost, but the problem arises due to its light structure makes it easily washed out after or during the adsorption process. This research provides a method to combine powdered activated carbon with clay and modify them into ceramic adsorbent, the combination is molded into small balls and then baked in a furnace at a high temperature of 1000 °C.

Clay has been well known as the low-cost adsorbent for pollutants removals such as metal ions, organic pollutants and bacterias in water purification because it is inexpensive due to its wide availability and a good adsorber [24]-[26]. Clay is a good adsorbent because of its chemical and mechanical stability, large specific area, layered structure, and high cation exchange capacity [26], [27]. However, raw clay has low adsorption of some anionic pollutants regarding their negative charges on the surface [28]. For example, clay minerals such as kaolinite, montmorillonite, and illite are able to adsorb arsenate in the range of 0.15 to 8.4 μmol/g, it is lower than iron containing minerals such as ferrihydrite and goethite which can adsorb arsenate in the range around 200 μmol/g to 700 μmol/g [29]. Thus, raw clays can be mixed with other metal ions like irons as a new modification to improve the adsorption capacity [30], [31].

Adsorption is the process in which matter is extracted from one phase and concentrated at the surface site of a second phase (interface accumulation). This is a surface phenomenon as opposed to absorption where matter changes solution phase, e.g. gas transfer.

Activated carbon and clay have widely used in industry and in many other applications to capture unwanted pollutants like heavy metal ions and groups in fluids etc. Carbon material with enhanced surface properties has been proved as an excellent adsorbent.

Activated carbon has a huge surface area. The interior of surface area is mostly the micro- and macropores. The usual range of surface area is 300-1500 m^2/gram.

Quality and hardness of the activated carbon are a function of the starting material and the activation process. If we have to remove soluble material from the solution phase, but the material is neither volatile nor biodegradable, we often conduct adsorption processes of electrostatic due to charged sites on the surface. The adsorption process goes up along with ionic charge but the hydrated radius usually goes down. pH often gives great impact on the surface charge of the adsorbent also the charge on the solute. Generally, for organic material as pH goes down adsorption goes up.

Adsorption using activated carbon as the adsorbent is commonly carried as three successive steps. The first step is film transport, where the solute is transported through the stagnant boundary layer onto the activated carbon particle, the second is pore diffusion, where the solute is transported through the pores within activated carbon, and the third is the actual adsorption, where the solute is attached to the activated carbon surface sites. The rate of solute adsorption can be limited by one or more of these steps. In many cases, the process is limited by the actual adsorption.

Since activated carbon is relatively expensive, adsorption would not be feasible unless the carbon can be regenerated after exhaustion. Spent carbon is usually regenerated at 500 °C under low oxygen conditions in the presence of steam.

Activated carbon loss is about 5-15% for each regeneration. Adsorbed organics are volatilized and oxidized during the regeneration process.

Investigation of heavy metallic ions (Al and Mn) on adsorption capacity contained in acid mine drainage is the aim of this research, by modifying activated carbon and clay into ceramic adsorbent with respect to the variation of adsorbent ratio, contact time and pH, the evaluation of its adsorption capacities were under batch experiments. A simple activation mechanism is performed with NaOH 48% solution to enhance the adsorption process.

II. MATERIAL AND METHOD

A. Material

Natural clay in this study was collected from a site in the Palembang City of Indonesia. It was washed with deionized water, dried at 100 °C for 1 h and crushed into a powder with mortar.The dried clean clay with the desired particle size was kept in a dry and clean container for further processing.

Ten adsorbent samples were prepared by varying the ratio of activated carbon:clay (2:38, 3:37, 4:36, 5:35, 6:34, 7:33, 8:32, 9:31, 10:30, 11:29) with total adsorbent mass of 1000 g.

First, the powdered carbon and clay were mixed and stirred until carbon was evenly mixed with clay, add enough deionized water to the mixtures and mold into small balls approximately 5 mm diameter. Afterwards, these balls were furnaced at 1000 °C for 48 h. Finally, the small balls had become ceramics and were ready for use.

B. Method

The pore systems usually become inaccessible with increasing deposits which block the pore systems. These may cause the pores become smaller and prevent the build-up reactions inside the pores so that the activation treatment should be carried on.

Activated carbon ceramic balls were soaked with NaOH 48% for 1 h to remove deposited unwanted materials inside the pores. After that, the deionized water was used to wash the activated carbon ceramic. Finally, the activated carbon ceramic was dried at 150 °C in an oven for 1 h.

For the adsorption experiments, the optimum adsorbent loading was found to be 700 g/500 ml. Various contact time (30, 60, 90, 120, 150, 180, 210, 240, 270, 300 min) and carbon:clay ratio (2:36 - 11:29) to study the effect of activated carbon ceramic adsorbent on the adsorptive removal of heavy metal pollutants in acid mine drainage. Further experiments are to investigate pH of acid mine drainage after adsorption process in order to expose the effect of heavy metal removal to acid mine drainage pH. The contacts between adsorbent and adsorbate were conducted in a glass column with 50 cm height and 5.2 cm diameter. The adsorbate was pumped with 2 l/min flow rate from a feed container to the top of the column using a small tube.

The concentration of Al and Mn of liquid samples before and after treatment with adsorbent were carried out by atomic absorption spectrometry (AAS). The equation for calculating adsorption capacity is:

$$q_e = \frac{[C_o] - [C_t]\, V}{W} \tag{1}$$

and the percentage of adsorption is measured by equation:

$$\% \ q_e = \frac{[C_o] - [C_t]}{[C_o]} \times 100\% \qquad (2)$$

where q_e is the adsorption capacity (mg g^{-1}), C_o and C_t are the initial and equilibrium concentration of metallic ions in the samples solution (mg L^{-1}). V is the volume of testing solution (L) and W is the weight of adsorbent (g) [32], [33].

III. RESULTS AND DISCUSSION

As we can see in Fig. 1, each contact time has its own performance characteristic on heavy metallic ions capture. The longer contact time causing the higher removal of heavy metallic ions. The initial concentration of each metallic ions is 7.95 mg L^{-1} for Mn and 10.12 mg L^{-1} for Al. Mn adsorption capacity increases from 0.03 mg g^{-1} to 4.01 mg g^{-1} and Al adsorption capacity increases from 0.8 mg g^{-1} to 6.57 mg g^{-1} by contact time variation from 30 to 300 min. The most favorable result of Mn adsorption capacity percentage is 70.69% and Al is 90.91% on 300 min contact time. The residual concentrations of heavy metallic ions were analyzed by Atomic Absorption Spectrometry (AAS), heavy metallic ions at which species were tested only in the form of cations.
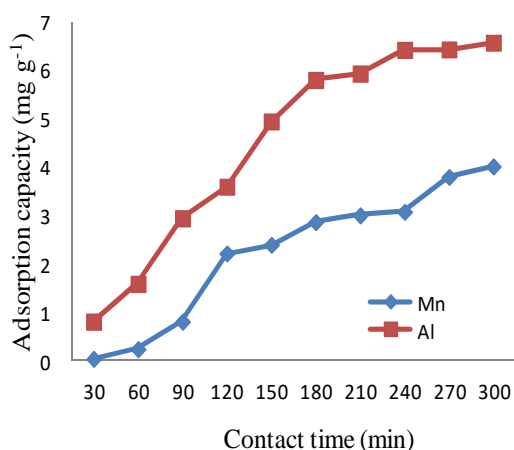

Fig. 1 Heavy metallic adsorption capacity with contact time variation

TABLE I
ADSORPTION CAPACITY ON VARIOUS CONTACT TIME

Contact time (min)	Adsorption Capacity (mg g^{-1})	
	Mn	Al
0	0,03	0,80
30	0,23	1,58
60	0,80	2,94
90	2,21	3,59
120	2,39	4,94
150	2,86	5,80
180	3,00	5,93
210	3,07	6,42
240	3,79	6,43
270	4,01	6,57
300	0,03	0,80

Fig. 1 and Table 1 have shown the effect of contact time on the adsorption capacity which has been studied. The adsorption capacity increases respectively. The adsorption data deriving the relationship between the adsorption ability and contact time. The longer contact time the higher adsorption capacity. Contact time has given enough access for the adsorbent to make bindings with more heavy metal cations (Mn and Al) and to make the binding sites on the adsorbent surface.

On Fig. 2 and Table 2 we can see that the adsorption capacity of each activated carbon:clay ratio increases, respectively. The initial concentration of each metallic ions is 7.95 mg L^{-1} for Mn and 10.12 mg L^{-1} for Al. The carbon:clay ratio plays an important role in adsorption capacity, Mn adsorption capacity increases from 0.02 mg g^{-1} to 4.11 mg g^{-1} and Al increases from 0.94 mg g^{-1} to 7.14 mg g^{-1} by adding activated carbon mass in an adsorbent ratio from 2 to 11. The most favorable result of Mn adsorption capacity percentage is 72.33% and Al is 98.81% at 11:29 adsorbent ratio. The addition of activated carbon mass contributes in improving adsorption capacity, it is assumed that its pore sizes are large enough for adsorbing other materials related to its porous characteristic resulting high adsorption capacity.
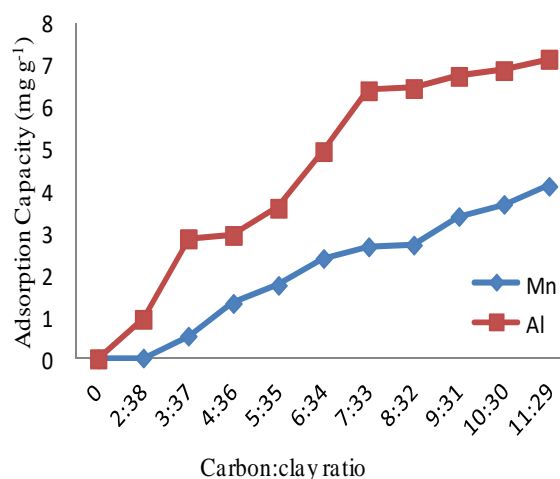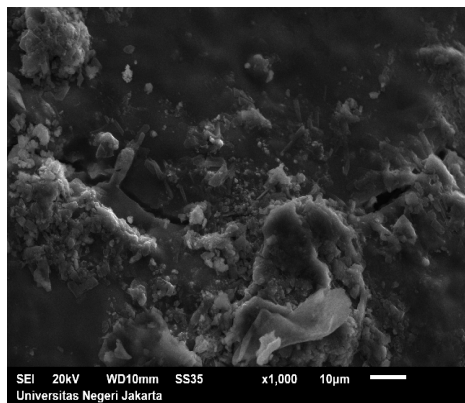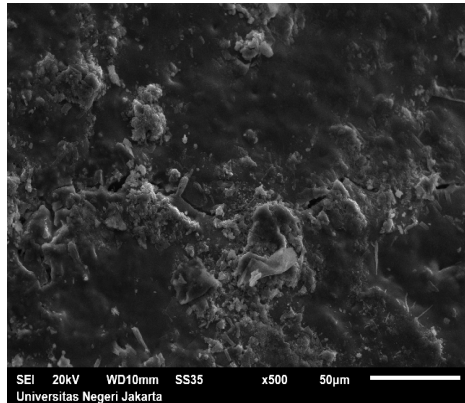

Fig. 2 Metallic ions adsorption capacity with carbon:clay ratio variation on contact time 300 min

TABLE II
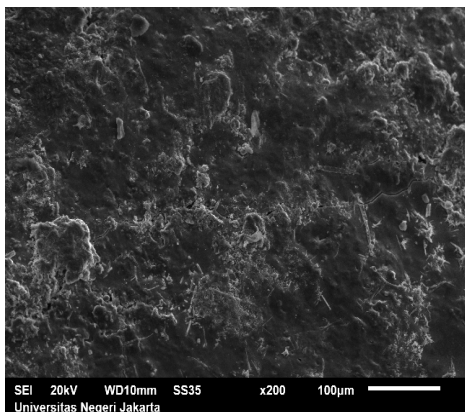ADSORPTION CAPACITY OF VARIOUS CARBON : CLAY RATIO

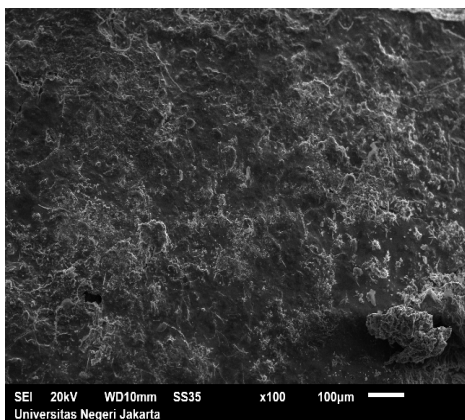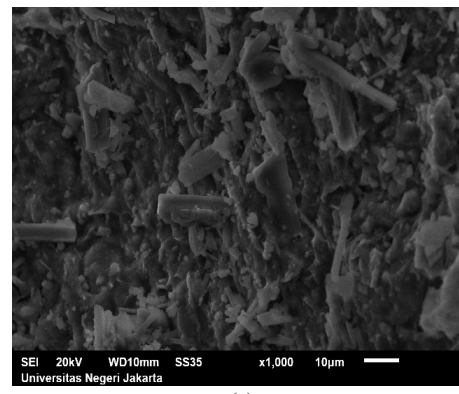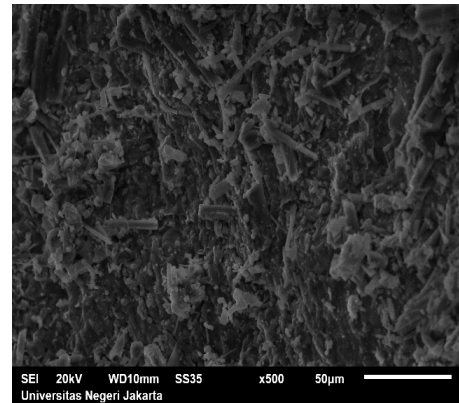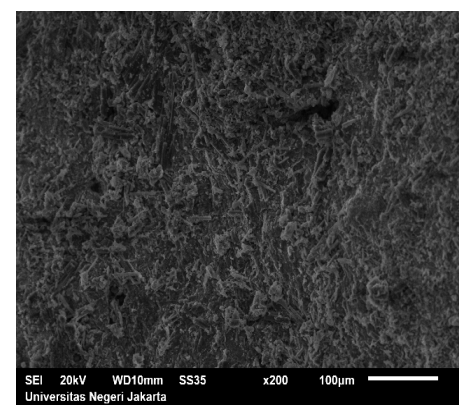| Carbon : Clay Ratio	Adsorption Capacity (mg g^{-1})	
	Mn	Al
0	0	0
2:38	0,02	0,94
3:37	0,54	2,86
4:36	1,32	2,94
5:35	1,75	3,59
6:34	2,39	4,94
7:33	2,67	6,39
8:32	2,71	6,44
9:31	3,39	6,73
10:30	3,68	6,87
11:29	4,11	7,14

(a)

(b)

(c)

(d)

Fig. 3 (a-d) SEM images of adsorbents surface structure before adsorption process by 100-1000 time of magnification
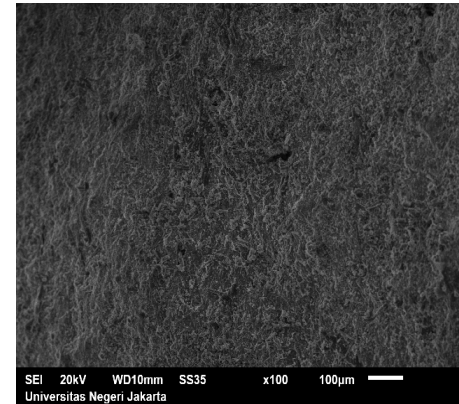

(a)

(b)

(c)

(d)

Fig. 4 (a-d) SEM images of adsorbents surface structure after adsorption process by 100-1000 times magnification

After the adsorption process, the surface area seems completely changed, by comparing images shown in Fig. 3 and Fig. 4 we can see that the surface pores have been covered almost evenly due to materials adsorbed and attached on to the entire area.

Most of the adsorbents with large surface areas have a large number of pores with different sizes and unique shapes. Based on the definition of the International Union of Pure and Applied Chemistry (IUPAC), there are three types of well-known pores: macro pore, mesopore, and micropore. The macro pore and the mesopore are mainly relevant to mass transfer processes into the inner-side of the adsorbent material, and the volume of micropore primarily determines the size of the surface internally also the adsorbent capacity. As stated, the internal surface area shall increase along with increasing the volume of micropore. In principle, when the micropore volume gets higher, the amount of adsorbate that can be adsorbed also gets higher. Regarding the adsorbent types, surface chemistry affects the adsorbate/adsorbent interaction. This is especially true for the adsorption of ions onto engineered adsorbents, but in some cases, it may also relevant for the adsorption onto activated carbons.

The adsorbed ions usually attached strongly and bound to a compact layer directly on to the surface. This first part of the layer is also referred to as the surface layer. As can be seen from the Fig. 3 and 4 that the adsorption of ions can lead to the change the surface complexes depending on the ion and the width of surface sites that involve in the adsorption process.

The model of surface area where ions may attach can also be considered as the surface sites by having bonds without losing their hydration water. It can be determined that the water molecule is placed between the ion and the adsorption site. Therefore, this causing wider distance to the surface and the binding strength is weaker compared to the inner side of the adsorbent material. The layer is also a part of the adsorbent material after adsorption process occurred. Beyond the layer, the surface layer ions charge oppositely to the ions charge adsorbed.
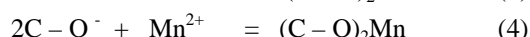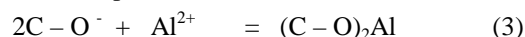
On Fig. 5 and Table III, it can be observed that the activation process has made pH increase respectively along with length of contact time, longer contact time causing higher pH increase, this is because activated carbon has alkaline groups on its surface, ion exchange process in aqueous solution during lead sorption at $6 < pH < 7.7$ explains the increasing pH and the sorption competition between H^+ and Pb^{2+} at $pH < 6$ [34].

In this research, static interaction of the cations, Mn and Al must take place during ion exchange process. In describing a sorption mechanism of Mn and Al with surface groups, either the form of surface groups or the form of heavy metallic cations must be taken account. The hydrolysis pH of heavy metallic cations is thought to be 4.0 and the precipitation pH is 7.1. When the activated carbon is basic, as in this case, the hydroxyl groups of the activated carbons are also in their basic form $C - O^-$.

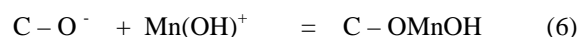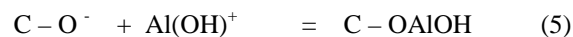The sorption process by ion exchange in the aqueous solution can be described in the following manner.

$$C - O^- + H_2O = C - OH + HO^- \qquad (1)$$
$$C - O^- + H^+ = C - OH \qquad (2)$$
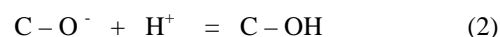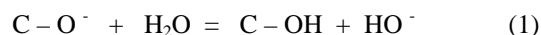
explaining the increasing pH and the sorption competition between Al and Mn at pH **4**.

$$2C - O^- + Al^{2+} = (C - O)_2Al \qquad (3)$$
$$2C - O^- + Mn^{2+} = (C - O)_2Mn \qquad (4)$$

explaining the Al and Mn sorption at pH 4.

$$C - O^- + Al(OH)^+ = C - OAlOH \qquad (5)$$
$$C - O^- + Mn(OH)^+ = C - OMnOH \qquad (6)$$

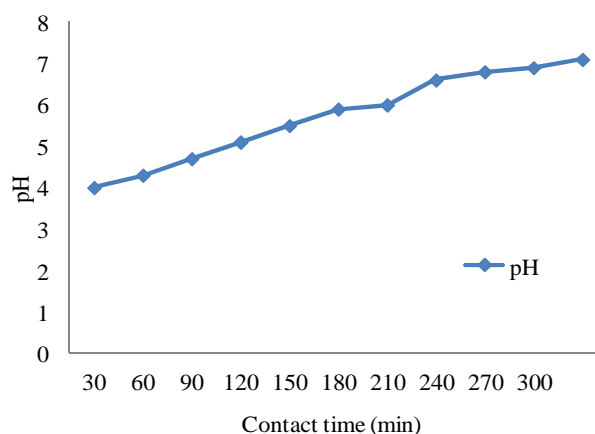explaining the Al and Mn sorption at $4 < pH < 7,1$.

Fig. 5 pH increase on contact time (min) variation (carbon:clay ratio 11:29)

TABLE III
PH ON VARIOUS CONTACT TIME (CARBON : CLAY RATIO 11:29)

Contact time (min)	pH carbon:clay ratio 11:29
0	4.0
30	4.3
60	4.7
90	5.1
120	5.5
150	5.9
180	6.0
210	6.6
240	6.8
270	6.9
300	7.1

Fig. 6 and Table 4 have shown that adsorption capacity has significantly increased after activation mechanism using NaOH 48%. This result indicates the activation process has to result the pore walls of adsorbent widened and clog free during the activation process resulting in the formation of new pores or converting to wider pores. After the activation stage, the surface has risen its adsorption area sharply and become exposed due to the activation mechanism gives addition in micropore volume. These are suitable for the activation purpose.
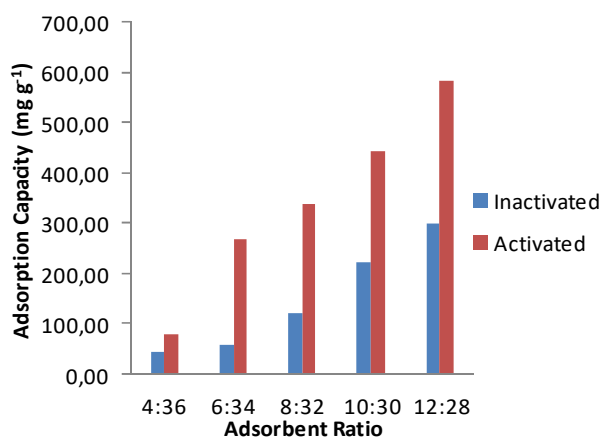
Fig. 6 The effect of activation mechanism using NaOH 48% on adsorption capacity

TABLE IV
ADSORPTION CAPACITY OF VARIOUS CARBON : CLAY RATIO BETWEEN
INACTIVATED AND ACTIVATED ADSORBENTS

Carbon : Clay Ratio	Inactivated	Activated
4:36	42,12	78,18
6:34	56,27	269,43
8:32	120,65	338,41
10:30	222,21	442,15
12:28	301,12	582,18

IV. CONCLUSION

Adsorbent ratios between activated carbon and clay as adsorbent materials had given chemical and physical properties that greatly affected heavy metallic ions (Mn and Al) removal performance and pH increase. The higher activated carbon mass in adsorbent material causing higher removal capacity. Evidently, the characteristic of adsorbent materials had improved adsorption capacity performance. The effect of contact time on the adsorption capacity had also been analyzed where it fairly increased respectively along with longer contact time. This research had proved that the adsorbent used was effective enough to reduce heavy metals pollutant in acid mine drainage, its adsorption capacity percentage reached most favorable results of 72.33% for Mn and 98.81% for Al at activated carbon:clay ratio 11:29 on contact time 300 min, also pH increase from 4.0 to 7.1. Accordingly, it was concluded that activated carbon ceramic could be a recommended adsorbent to solve the acid mine drainage problem.

NOMENCLATURE

q	adsorption capacity	mg g^{-1}
Co	initial concentration	mg L^{-1}
Ct	equilibrium concentration	mg L^{-1}
V	volume of testing solution	L
W	weight of adsorbent	g

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