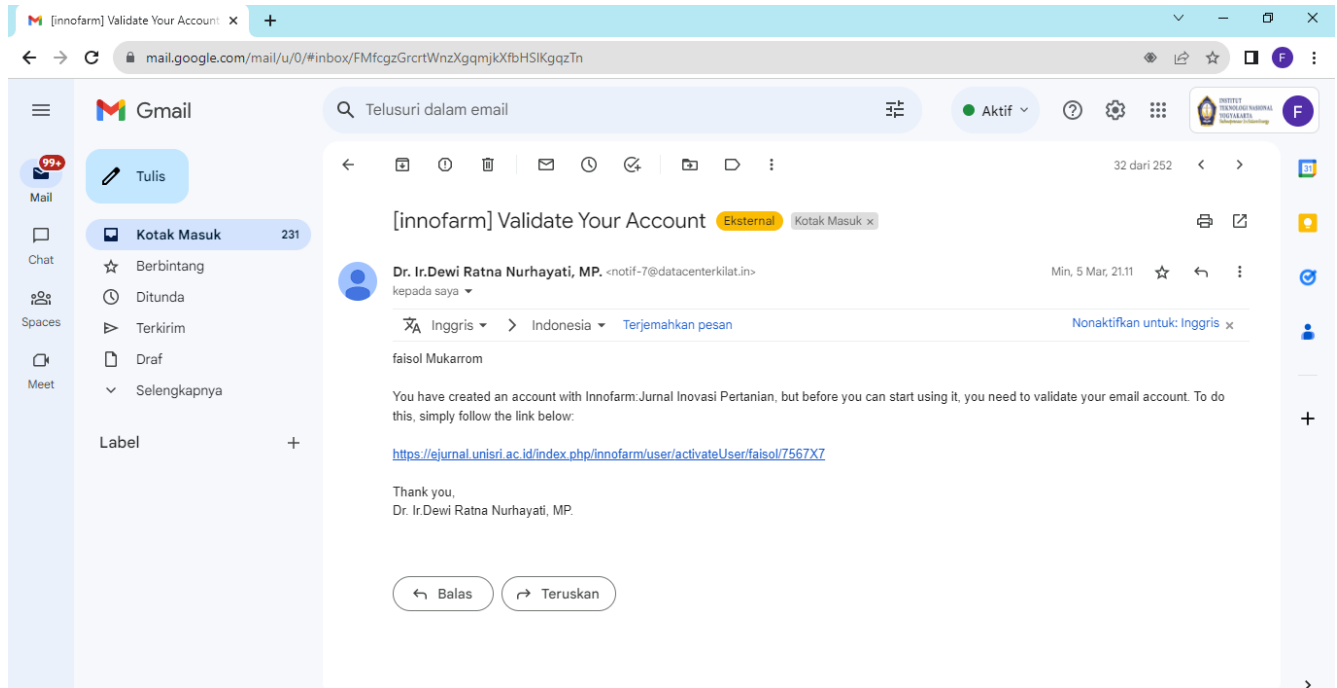## Account Validation

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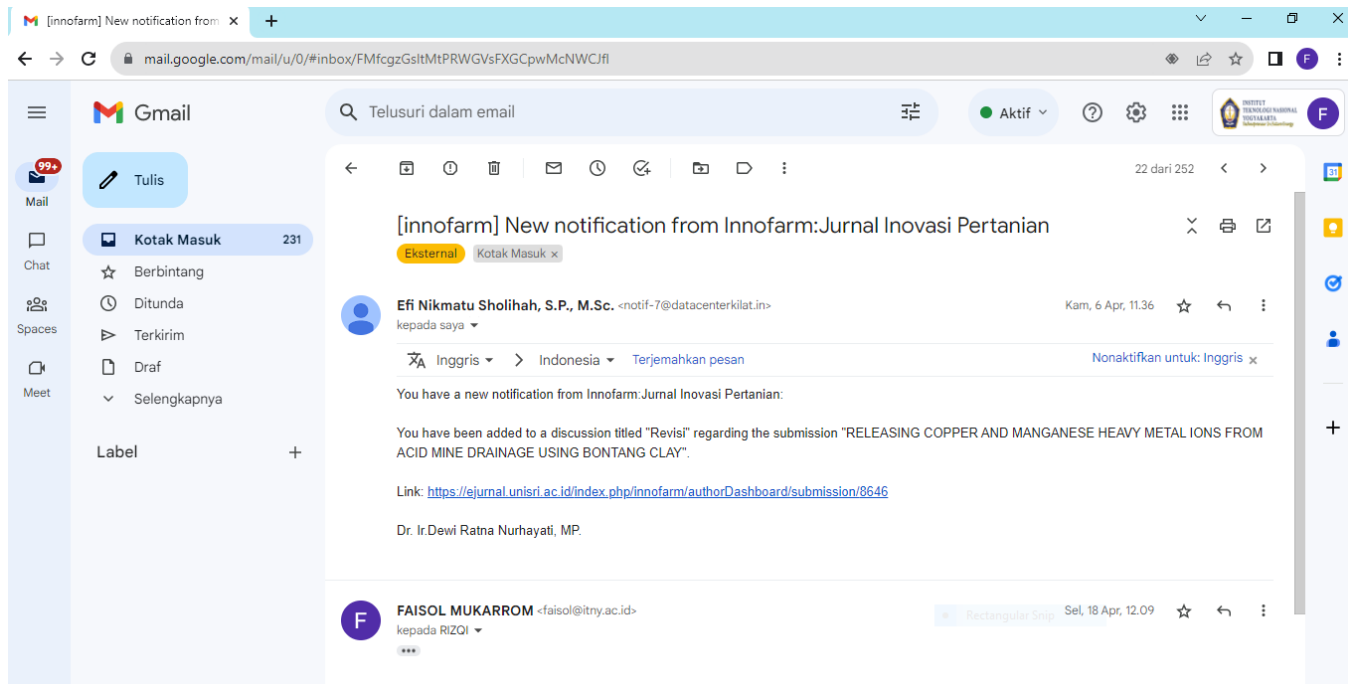
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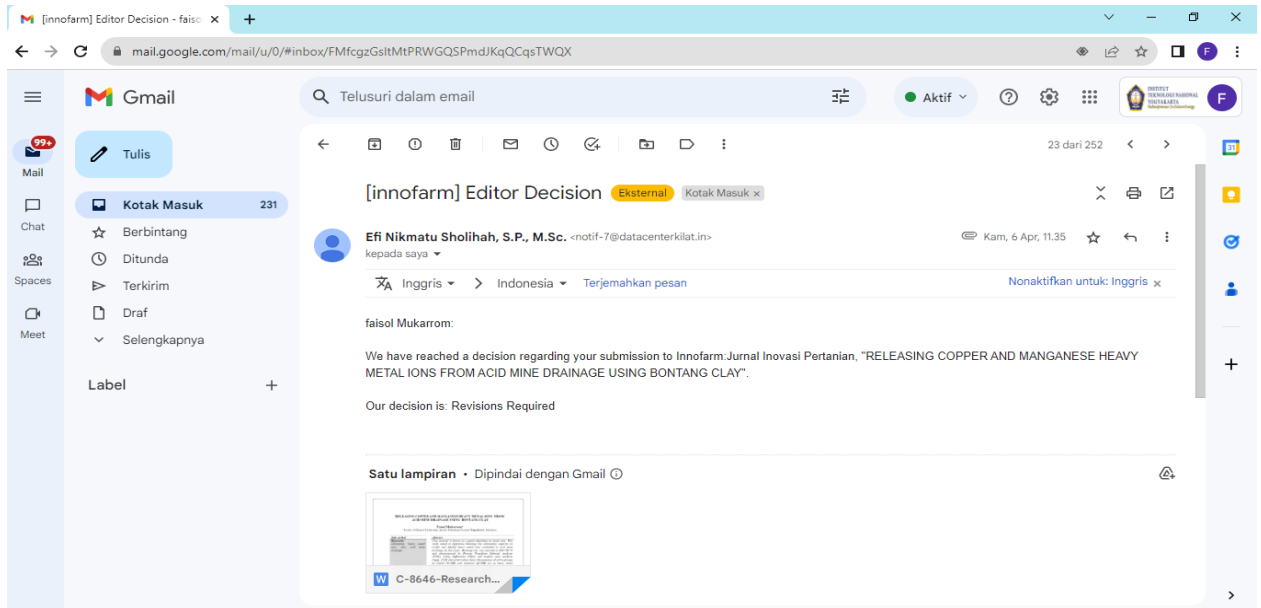
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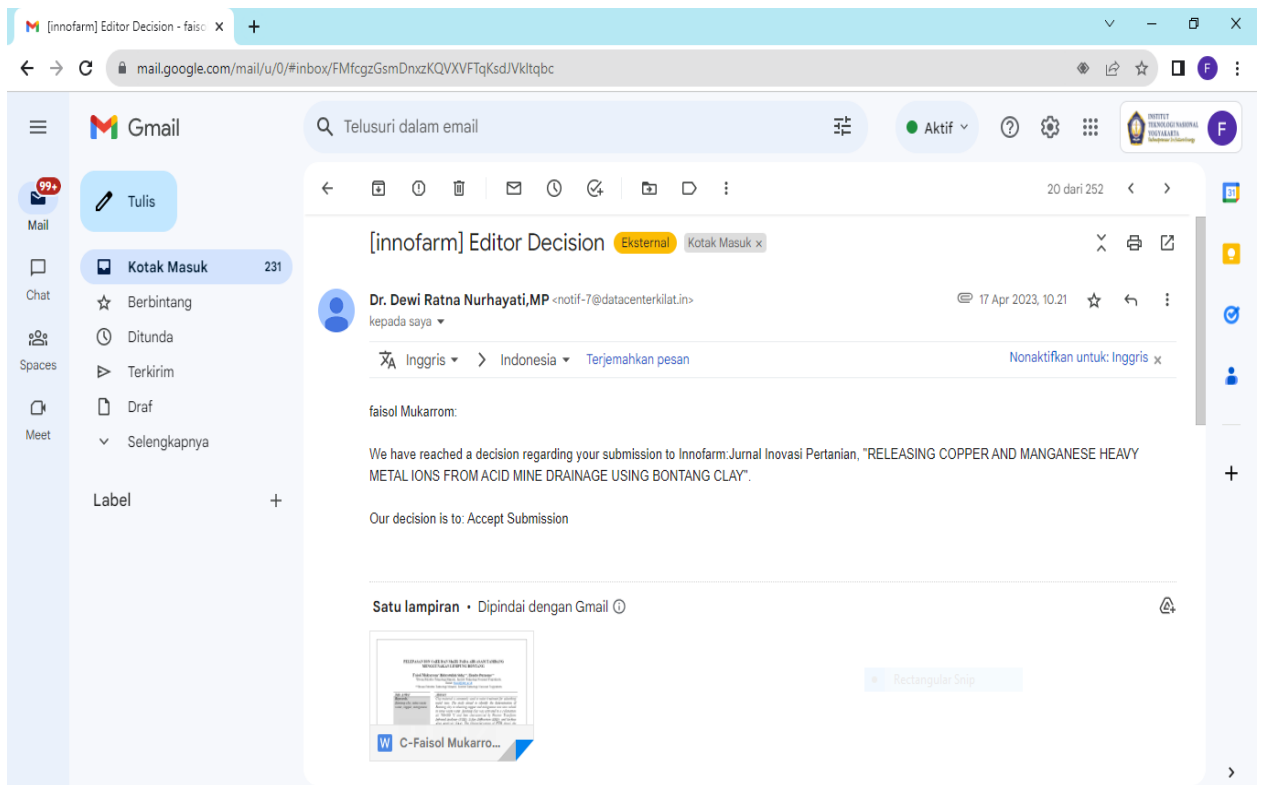
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RELEASING *COPPER* AND *MANGANESE* HEAVY METAL IONS FROM ACID MINE DRAINAGE USING BONTANG CLAY

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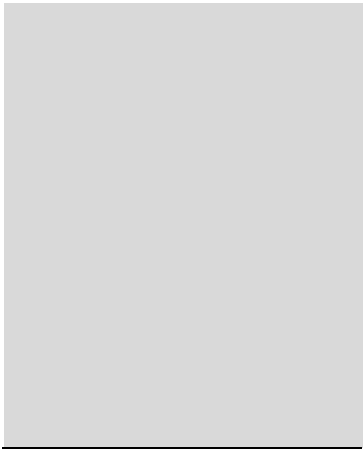| Info Artikel	Abstract
Keywords: adsorption, heavy metal ions, clay, acid mine drainage	*Clay mineral is known as a good adsorbent on metal ions. This study aimed to determine Bontang clay adsorption capacity on Cu(II) and Mn[II] heavy metal ions contained in acid mine drainage. In this study, Bontang clay was calcined at 600-700 ºC and characterized by Fourier Transform Infrared Analyzer (FTIR), X-Ray Diffraction (XRD), and Surface Area Analyzer (SAA). FTIR characterization shows the presence of active groups of silanol (Si-OH) and aluminol (Al-OH) act as heavy metal adsorbent. XRD characterization demonstrated the presence of montmorillonite, kaolinite, illite and quartz. SAA characterization revealed that after activation was greater than before activation, in amount of 34.793 m^2/g for pre-activation and 49.859 m^2/g for post activation. Calcination of Bontang clay improved the adsorptive capability of Cu[II] and Mn[II]. Maximum adsorption percentage of Cu[II] on raw and calcined Bontang clay were 96,53% and 94.67% at initial concentration of 10 mg/dm^3 and they were 74.91% and 32.82 % for Mn[II. They decreased to 7.2% and 8.3% for raw and calcined Cu[II] and 4.3% and 6.8% for Mn[II] at initial concentration of 5,000 mg/dm^3. The higher adsorption percentage is for lower concentrations for both Cu[II and Mn[II].*
	Abstrak
Kata kunci: Adsorpsi, ion logam berat, lempung, air asam tambangpetunjuk penulisan; jurnal pertanian; template artikel	Mineral lempung dikenal sebagai adsorben yang baik pada ion logam. Penelitian ini bertujuan untuk mengetahui daya adsorpsi lempung Bontang terhadap ion logam berat Cu(II) dan Mn[II] yang terkandung dalam air asam tambang. Pada penelitian ini tanah liat Bontang dikalsinasi pada suhu 600-700 ºC dan dikarakterisasi dengan Fourier Transform Infrared Analyzer (FTIR), X-Ray Diffraction (XRD), dan Surface Area Analyzer (SAA). Karakterisasi FTIR menunjukkan adanya gugus aktif silanol (Si-OH) dan aluminol (Al-OH) yang berperan sebagai adsorben logam berat. Karakterisasi XRD menunjukkan adanya montmorillonit, kaolinit, ilit dan kuarsa. Karakterisasi SAA menunjukkan bahwa setelah aktivasi lebih besar dibandingkan sebelum aktivasi, yaitu

sebesar 34,793 m2/g untuk pra aktivasi dan 49,859 m2/g untuk pasca aktivasi. Kalsinasi lempung Bontang meningkatkan daya serap Cu[II] dan Mn[II]. Persentase adsorpsi maksimum Cu[II] pada tanah liat Bontang mentah dan terkalsinasi adalah 96,53% dan 94,67% pada konsentrasi awal 10 mg/dm3 dan Mn[II sebesar 74,91% dan 32,82%. Mereka menurun menjadi 7,2% dan 8,3% untuk Cu[II] mentah dan terkalsinasi dan 4,3% dan 6,8% untuk Mn[II] pada konsentrasi awal 5.000 mg/dm3. Persentase adsorpsi yang lebih tinggi adalah untuk konsentrasi yang lebih rendah untuk Cu[II dan Mn[II].

INTRODUCTION

Indonesia has up to 105.2 billion metric tons of coal resources, which is approximately 21.1 billion metric tons are in reserves (Sulistijo et al, 2013). Large scale exploitation of coal in Indonesia experienced a rapid resurgence by the operating of new mines in the 1980s, about 20 years after the investment and mining laws was liberated (Friederich and Leeuwen, 2017). Coal mining is one of the major industries which has a significant contribution to the economic development of a country, however it also causes serious deterioration to the environment (Dutta et al, 2017; Ranjan, 2019; Pranoto et al, 2017)

Most of the mining operations in developed countries apply the open pit methods (Oggeri et al, 2019). The big amount of overburden as waste in open pit mining needs a modern management due to the complexity within the handling and the materials (Jabłonska and Siedlecka, 2015; Oggeri et al, 2019). Overburden loosening and disposal handling management has crucial impacts and should be very well organized considering both mining operation and reclamation ((Dutta et al, 2017; Oggeri et al, 2019).

The environmental impact commonly occurs in coal mine operation due to the outflow of highly acidic and heavy metals contained in water from mine areas generally termed as Acid Mine Drainage (AMD) (Jabłonska and Siedlecka, 2015; Ojonimi et al, 2019; Simate et al, 2014). Acidic solutions can be formed in decomposition process of sulfide minerals through oxidative process in waste practice in waste water (Bwapwa et al, 2017).

Heavy metals are commonly found in surface and subsurface water flows as well as in land filled leachates (Musso et a;, 2014; Abidi, 2019). Acid mine drainage is divided into three types i.e acid, neutral and basic drainages (Masindi, 2017). High concentration of heavy metals in AMD affects the environmental damage characterized by contaminated ecosystem due to its capacity of leaching and the existence of active bacteria (Bwapwa et al, 2017).

Main concern of mine impact is on AMD due to its low pH (pH 2-3) and the increasing of heavy metals ion concentrations such as Fe, Al, Mn, Zn, Ni, Co, Cd, Cu, Hg and Pb (Masindi, 2017; Choudhury et al, 2017). The neutral mine drainages are shown by neutral pH, low Fe concentration and high levels of dissolved arsenic (As), cadmium (Cd), lead (Pb), zinc (Zn) and others. The treatment application and methodology are simply deliberated (Lee at al, 2018).

Clay is a widely deposited material in the world, cheap and easily found (Pranoto et al, 2017; Lee et al, 2018). Some of this material origin are carboniferous clays layered accompanying the coal beds in many coal basins spread all over the world (Jabłonska and Siedlecka, 2015; Offeddu et al, 2015). Previous studies reported *Bontang clay* could be used as heavy metals adsorption media of water solutions (Jabłonska and Siedlecka, 2015).

MATERIAL AND METHODS

The sample of Bontang clay were taken from overburden of a coal mine area at Bontang East Kalimantan.

Preparation of Adsorbent

The sample of clay was cleaned from impurities and then milled down and screened into the grain size below 200 mesh. The samples were then desiccated in dry air[3]. This study of heavy metals Cu(II) and Mn(II) adsorption from aqueous solutions was done on suspensions of Bontang clay at a concentration of 2%. The 2% aqueous solutions of Cu(II) was poured into *Bontang clay* suspension with the initial concentrations of 10, 50, 100, 250, 500, 1000, 2500 and 5000 mg/dm[3].

Activation of Adsorbent

Adsorptive capability of an adsorbent was increased by physical activation. The increment was measured. The fine material was inserted into a cold muffle furnace (Thermoyne type 48000) by oxidation at a temperature of about 600^0-700^0C for 2 hours (Jabłonska and Siedlecka, 2015; Alaistara et al, 2018). The furnace was then switched off and the activated material was left inside until the temperature is cooled down. The same adsorption process was also carried out in raw Bontang clay.

Application of Adsorption

The adsorption study of Bontang clay on heavy metals were taken on synthetic solutions made based on analytically pure compounds of copper and lead containing one of these metals ions whose initial concentration similar to their concentration in acid mine drainage.

This study was done on suspensions of clay having concentration of 2%. The suspensions were poured with heavy metals solution of initial concentrations of 10, 50, 100, 250, 500, 1000, 2500 and 5000 mg/dm[3]. The samples then were stirred with a rotational vibrator using *hot plate + stirrer* (Thermolyne type 1000 *Stirrer plate)* for about two hours at the temperature of 20°C and then placed in a dark room for 22 h. Next, the solution was poured and the sediment was removed by a centrifugal clarifier at 2500 rpm.

The mass of adsorbed metal per unit of sediment mass, q, was calculated with the formula as follows (Jabłonska and Siedlecka, 2015).

$$q \text{ ¼ } \frac{C_i - C_e}{} V \frac{\Sigma_{mg}\Sigma^3}{mg} \qquad (1)$$

Note: C_i, C_e e initial and equilibrium concentrations of Cu(II) (mg/dm[3]), V e solution volume (dm[3]), m_e mass of used sediments (g). Three measurement were doing for each sample type and the mean values were taken for further calculations.

Adsorption Analysis

Adsorption Isotherms

Adsorption isotherm reflects the relationship the amount of adsorbate released from the liquid phase by unit mass of adsorbent at a same temperature(Hasan et al, 2019). The amounts of adsorbed metals were found and the adsorption isotherms q f(C_e) for Cu and Mn on the raw and calcined clays were determined. Freundlich and Langmuir adsorption isotherms were used in this study since they are very commonly used in quantitative description of adsorption. They describe a relation between the mass of adsorbate per unit mass of the adsorbent, also named as the adsorption capacity (q, mg/g), and the adsorbate concentration in the fluid in equilibrium (C_e, mg/dm[3]). Freundlich adsorption isoterm formula has the following form:

$$q \text{ ¼ } K_F C^n \text{ ð} mg=g\text{Þ}; \qquad (2)$$

where K_F and n are empirical constants depends on the adsorbent and adsorbate at a certain temperature.

Coefficient K_F is measured adsorption capacity.

Parameter n, where $0 < n < 1$, shows the intensity of adsorption. The curves relating to small values of n grow abruptly for small concentrations and slowly for greater ones, reflecting roughly equal adsorption for all the whole range of examined concentrations.

The formula of Langmuir adsorption isotherm is as follows:

$$1/Q \quad Q\frac{K_L C_e\_ðmg = g Þ;}{1 þ K_L C_e} \qquad (3)$$

where K_L and Q are constants for adsorbate and adsorbent at a certain temperature. Coefficient Q (mg/g) is the adsorption capacity, to when q tends for large C_e. Constant K_L (dm^3/mg), refers to as the Langmuir adsorption constant, reveals the strength of adsorbate and adsorbent interaction. Greater values of K_L result in curves sharply growing for small concentrations and mostly constant at a level of Q for others. Therefore, the greater value of K_L the lower concentration at when the the adsorbent were saturated.

When the Langmuir equation is compared with the Freundlich equation, both Q and K_F are s factors reflecting the adsorption capacity, and both K_L and n figure the shape of the isotherm. However, the Freundlich isotherm shows as unlimited values of q for large C_e, whereas the Langmuir isotherm is quite more realistic and gives q limited by Q. Coefficients K_F, n, Q, K_L, K_E and q_m are commonly determined from linearized forms of the isotherm equations. By taking logarithm of both sides of the Freundlich equation one obtains

$$\log q \ ¼ \ n \log c_e \ þ \ \log k_f; \qquad (4)$$

where $\log q$ is linear function of $\log C_e$ with coefficients $\log K_F$ and n. The linearized forms lead us to use the linear regression method. The Freundlich and Langmuir adsorption isotherms show similar value of the coefficient of determination. This show that the adsorption on the *Bontang clay* has a mixed nature and is non-uniform and nonspecific, which probably originates from non-homogeneity of the clay. Adsorption sites could differ in energy considerably, depends on whether they are located on an edge or in a defect position.

RESULT AND DISCUSSION
Identification of Adsorbent
FTIR Characterization

The FTIR spectrum of Bontang clay is shown on Figure 1. In pre and post activation, *Bontang clay* underwent changes on wave number shifts of Si-O-Si /Al-O-Al (from 1031 cm^{-1} to 1033 cm^{-1}) and Al-OH (from 912 cm^{-1} to 916 cm^{-1}).

Refer to the study carried by (Bwapwa et al, 2018), calcination makes changes as figured by increasing the intensity due to removal of impurities (Table 1).

Tabel1. FTIR adsorption of Bontang cla*y* pre- and post-activation

No	Functional Group	Wave number (cm^{-1})		
		References	Pre-activation	Post-activat2on
1	Al-OH-Al	3616	3695; 3623	3699; 3625
2	-OH	3500-3400	3421	3426

3	H-O-H	1639	1628	1635
4	Si-O-Si / Al-O-Al	1039	1002	1012
5	Al-OH	920-690	920	928
6	Si-O / Al-O	470,6	455	455

X-Ray Diffractometry Characterization (XRD)

XRD pattern of *Bontang clay* is shown on Figure 1. *Bontang clay* XRD analysis pre- and post-activation obtained the presence of mineral contents such as *kaolinite* (K), *montmorillonite* (M), *illite* (I), and *quartz* (Q), demonstrated by emergence of diffraction peaks on 2θ. In pre-activated *Bontang clay*, *kaolinite*, *montmorillonite*, *illite* and *quartz* appeared at 35.02º and 32.09º; 5.34º, 19.85º, and 61.65º; 27.70º; and 67.29º respectively. In activated Bontang clay, *Bontang clay*, *kaolinite*, 2*montmorillonite*, *illite* and *quartz* appeared at 30.24º, 39.35º, 60.2º; 5.65º, 19.10º, and 62.05º; 26.89º; and 64.37º respectively. Pre- and post-activation diffractogram demonstrates decrease on intensity and new peaks. Decrease intensity of each mineral shows the change of structure from crustal to amorphous.
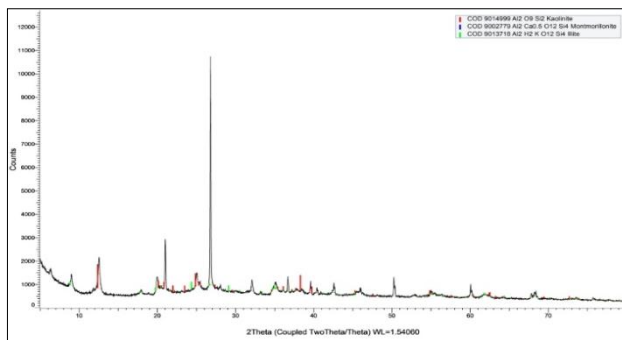

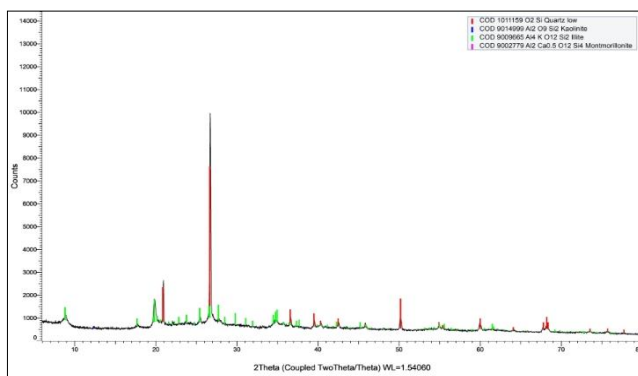**Figure 1.** XRD pattern of Bontang clay before activation

Figure 2. XRD pattern of Bontang clay after activation

Analysis of Surface Area

Surface area was obtained by presence of interaction between a solid substance and substances surrounding it, such as liquid and gas. The most important thing in determining size of surface area

is the molecular gas absorbed. Surface area can be analyzed by using N_2 adsorption-desorption test by using Brunauer, Emmet, and Teller (BET) equation.

In this study, pre-activation *Bontang clay* had specific surface area of 34,793 m^2/g while post-activated *Bontang clay* had specific surface area of 49,859 m^2/g. This surface area enlargement was possibly due to calcining process. This enlarged surface area provides an area for adsorbent's surface so that metals ion can be adsorbed.

Adsorption Analysis
Copper
The static adsorption on the claya resulted changes in Cu(II) concentration displayed in Figure 3 as below
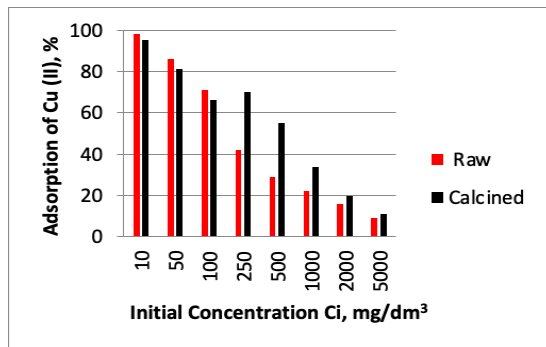

Figure 3. Adsorption percentage of Cu(II) on Bontang clay

There were decreases of Cu(II) adsorption percentage in all higher concentration as shown in Figure 4. The maximum adsorption percentage of raw *Bontang clay* on Cu[II] was 96.2 % for an initial concentration of 10 mg/dm^3 and 94.5% activated one. It downs to 42.6% for 250 mg/dm^3 and 70.2% for the calcined one. At the initial concentration of 5000 mg/dm^3, it equaled 8.3% for the raw *Bontang clay* and about 7.2% for the activated one.

The tested *Bontang clay* have Cu(II) adsorptive capabilities equal to or better than other adsorbents.
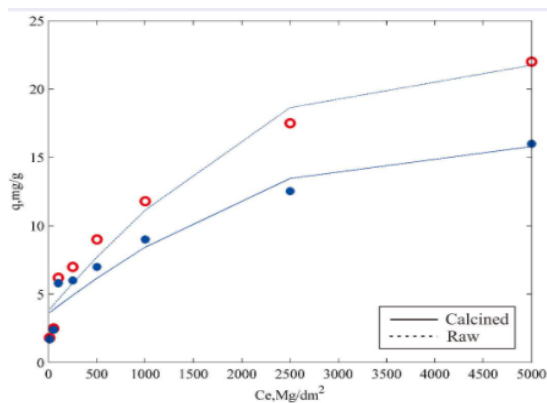

Figure 4. Adsorption isotherms of Cu(II) from *Bontang clay*

Manganese
The adsorption percentage of Mn ions depends on the initial concentrations. It is getting lower with higher initial concentration of Mn in the examined Bontang clay.

The adsorption percentage of Mn ions depends on the initial concentrations. It is getting lower with higher initial concentration of Mn in the examined Bontang clay.

As shown in Figure 5, for the raw *Bontang clay* it lowering from 74,62% for an initial

concentration of 10 mg/dm^3 and 76.67% for calcined one. At initial concentration of 250 mg/dm^3 they decreased to 32.65% and 44.29% for pre- and post-calcination respectively. The initial concentration of 5000 mg/dm^3, they were 4.33% for the raw *Bontang clay* and 6.87% for activated one.

Interpacket water and OH$^-$ groups on the surface of aluminosilicate was mostly removed by the activation of *Bontang clay*. This made in decreasing the number of negatively charged active sites on the metal surface.

A lowering in Mn(II) adsorption due to release of acid groups has also been studied in the case of active carbons activated at a temperature of about 600 $^\circ$C, although the carbons remain in a major part of surface non-acidic groups.

The study shows that the examined *Bontang clay* can adsorb metals in large amounts.
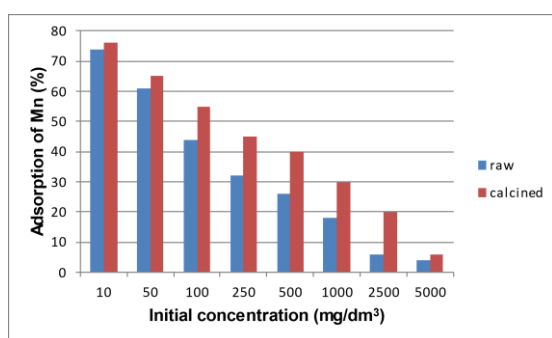

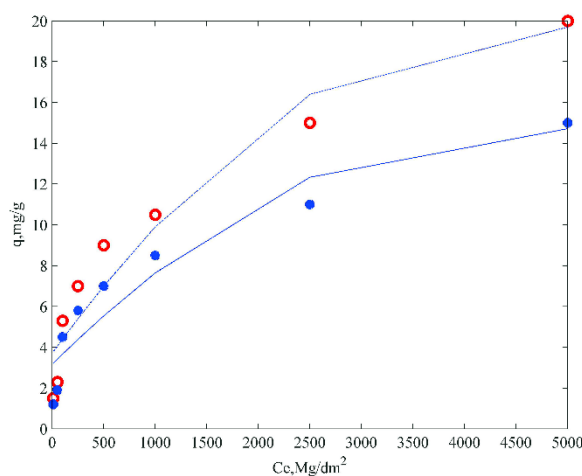**Figure 5.** Adsorption percentage of Mn(II) on *Bontang clay*

Figure 6. Adsorption isotherms of Mn(II) from *Bontang clay*

Adsorption Summary

Metals of Cu[II] and Mn[II] adsorbed by the examined *Bontang clay* satisfies approximately relation Cu(II) > Mn(II). The adsorption isotherms for Cu(II) and Mn(II) (Fig. 5 and 6) shows that the adsorption capacity are not more 5 mg/g if the concentration is less than 100 mg/dm^3.

The adsorption intensity rises gradually for Cu(II) and Mn(II) when the concentration were more than 250 mg/dm^3. The weak adsorption of Mn[II] on the *Bontang clay* could be related to the presence of small amount of fine pyrite the raw samples, but not exists in the XRD patterns. The presence of pyrite oxidation in clay suspensions are hydrolyzed leading in the occupation of exchange sites in the clay minerals by hydrogen ions, which compete with metal ions, significantly decreasing the adsorption intensity.

Activated *Bontang clay* shows very good adsorptive capabilities Mn[II]. However, the calcination did not make any significant changes in the crystalline structure for adsorption of Cu[II]. The adsorption isotherms reflect that the adsorbent was not fully saturated with the metal ions for high concentrations of metal ions relatively.

The results of the study show that the efficiency of releasing heavy metals cations on the raw and calcined *Bontang clay* depends on the structure of the clay and varies for each metal.

CONCLUSION

The results of the taken analysis leads to the following conclusions:

1. *Bontang clay* can be utilized as an adsorption media of Cu(II) and Mn[II] metal ions in AMD. It has been proven by this study, which obtained characterization of the adsorbents and adsorption percentage.
2. Calcination of *Bontang clay* at 600-700°C significantly improved the adsorptive capabilities for Cu(II) and less for Mn[II].
3. Copper ions adsorption capability of *Bontang clay* is better than its Manganese ion adsorption capability.
4. It is easy to obtain *Bontang clay* in coal mining areas, therefore. it is effective to lower the cost of acid mine drainage removal process.
5. The adsorption percentage of Cu(II) and Mn[II] by the aqueous *Bontang clay* solution increased with lowering concentrations.
6. In this study, the best copper adsorption was by the activated Bontang clay at 5,000 mg/dm^3.

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Final Draft

PELEPASAN ION Cu[II] DAN Mn[II} PADA AIR ASAM TAMBANG MENGGUNAKAN LEMPUNG BONTANG

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Info Artikel	Abstract
Keywords: *Bontang clay, mine waste water, copper, manganese*	*Clay material is commonly used in water treatment for adsorbing metal ions. The study aimed to identify the determination of Bontang clay in releasing copper and manganese ions ions solved in mine waste water. Bontang clay was activated in a calcination att 700-800 ºC and then characterized by Fourier Transform Infrared Analyzer (FTIR), X-Ray Diffraction (XRD), and Surface Area Analyzer (SAA). The Characterization of FTIR shows the presence of active groups of silanol (Si-OH) and aluminol (Al-OH) act as metal ions adsorbent. XRD shows the montmorillonite, kaolinite, illite and quartz. SAA displayed that post-activation was wider than pre-activation at 34.793 m^2/g for pre-activation and 49.859 m^2/g for after activated. Physical activation of Bontang clay increases the adsorption capability of Cu[II] and Mn[II]. Ultimate adsorption of Cu[II] on raw and activated Bontang clay were 96,53% and 94.67% at beginning concentration of 10 mg/dm^3 and they were 74.91% and 32.82 % for Mn[II. They lowered to 7.2% and 8.3% for raw and activated Cu[II] and 4.3% and 6.8% for Mn[II] at beginning concentration of 5,000 mg/dm^3. The better adsorption percentage is for more little concentrations for both Cu[II and Mn[II].*
	Abstrak
Kata kunci: Lempung Bontang, air asam tambang, tembaga, mangan	Mineral lempung dikenal sebagai adsorben yang baik pada ion logam. Penelitian ini bertujuan untuk mengetahui daya adsorpsi lempung Bontang terhadap ion Cu(II) dan Mn[II] yang terkandung pada limbah air tambang. Pada penelitian ini tanah liat Bontang dikalsinasi pada suhu 600-700 ºC dan dikarakterisasi dengan Fourier Transform Infrared Analyzer (FTIR), X-Ray Diffraction (XRD), dan Surface Area Analyzer (SAA). Karakterisasi FTIR me adanya gugus aktif silanol (Si-OH) dan aluminol (Al-OH) yang berperan sebagai adsorben logam berat. Karakterisasi XRD menunjukkan adanya montmorillonit, kaolinit, ilit dan kuarsa. Karakterisasi SAA menunjukkan bahwa setelah aktivasi lebih besar

dibandingkan sebelum aktivasi, yaitu sebesar 34,793 m2/g untuk pra aktivasi dan 49,859 m2/g untuk pasca aktivasi. Kalsinasi lempung Bontang meningkatkan daya serap Cu[II] dan Mn[II]. Persentase adsorpsi maksimum Cu[II] pada tanah liat Bontang mentah dan terkalsinasi adalah 96,53% dan 94,67% pada konsentrasi awal 10 mg/dm3 dan Mn[II sebesar 74,91% dan 32,82%. Mereka menurun menjadi 7,2% dan 8,3% untuk Cu[II] mentah dan terkalsinasi dan 4,3% dan 6,8% untuk Mn[II] pada konsentrasi awal 5.000 mg/dm3. Persentase adsorpsi yang lebih tinggi adalah untuk konsentrasi yang lebih rendah untuk Cu[II dan Mn[II].

PENDAHULUAN

Indonesia memiliki sumberdaya batubara sebesar 105,2 miliar metrik ton, dimana sekitar 21,1 miliar ton diantaranya berklasifikasi cadangan (Sulistijo dkk, 2013). Eksploitasi batu bara skala besar di Indonesia mengalami peningkatan yang cepat dengan beroperasinya perusahaan-perusahaan tambang baru pada 1980-an, sekitar 20 tahun setelah undang-undang investasi dan pertambangan dibebaskan (Friederich dan Leeuwen, 2017). Pertambangan batubara memiliki peran signifikan pada pembangunan ekonomi suatu negara, namun juga menyebabkan kerusakan lingkungan yang serius (Dutta dkk, 2017; Ranjan, 2019; Pranoto dkk, 2017).

Sebagian besar operasi penambangan di negara maju menerapkan metode tambang terbuka (Oggeri dkk, 2019). Jumlah *overburden* yang besar sebagai limbah pada tambang terbuka memerlukan pengelolaan yang modern karena rumitnya penanganan dan materialnya (Jabłonska dan Siedlecka, 2015; Oggeri dkk, 2019). Manajemen pembongkaran dan pembuangan lapisan tanah penutup memiliki dampak penting dan harus diatur dengan sangat baik pada kegiatan operasi penambangan dan reklamasi ((Dutta dkk, 2017; Oggeri dkk, 2019).

Dampak lingkungan yang umumnya terjadi pada operasi tambang batubara akibat terarutnya logam berat yang terkandung dalam air dari area tambang umumnya disebut sebagai Air Asam Tambang (AAT) (Jabłonska dan Siedlecka, 2015; Ojonimi dkk, 2019; Simate et al , 2014). Larutan asam dapat terbentuk dalam proses dekomposisi mineral sulfida melalui proses oksidatif dalam pengelolaan air limbah (Bwapwa dkk, 2017).

Logam berat umumnya ditemukan pada aliran air permukaan dan bawah permukaan serta lindi yang terisi tanah (Musso dkk, 2014; Abidi, 2019). Air asam tambang dibagi menjadi tiga jenis yaitu asam, netral dan basa (Masindi, 2017). Konsentrasi logam; berat yang tinggi pada air asam tambang berdampak pada kerusakan lingkungan yang ditandai dengan ekosistem yang tercemar akibat kapasitas lindi dan adanya bakteri aktif (Bwapwa dkk, 2017).

Perhatian utama dampak tambang adalah pada AAT karena memiliki pH rendah (pH 2-3) dan peningkatan konsentrasi ion logam berat (Masindi, 2017; Choudhury dkk, 2017). AAT yang netral ditunjukkan oleh pH netral, konsentrasi Fe rendah dan kadar arsenik (As) terlarut yang tinggi, kadmium (Cd), timbal (Pb), seng (Zn) dan lain-lain. Aplikasi dan metodologi pengelolaannya hanya dibahas secara sepintas (Lee at al, 2018).

Lempung merupakan material yang banyak diendapkan di dunia, murah dan mudah ditemukan (Pranoto dkk, 2017; Lee dkk, 2018). Beberapa asal material ini adalah lapisan tanah liat karbon yang menyertai lapisan batubara di banyak cekungan batubara yang tersebar di seluruh dunia (Jabłonska dan Siedlecka, 2015; Offeddu dkk, 2015). Penelitian sebelumnya melaporkan tanah liat dari daerah tambang batubara dapat digunakan sebagai media adsorpsi logam berat dalam larutan air (Jabłonska dan Siedlecka, 2015).

METODE

Sampel lempung Bontang diambil dari *overburden* area tambang batubara di Bontang Kalimantan Timur.

Preparasi of Adsorben

Sampel tanah liat dibersihkan dari kotoran kemudian ditumbuk dan disaring menjadi ukuran butir di bawah 200 mesh. Sampel kemudian dikeringkan di udara kering. Kajian adsorpsi logam berat Cu(II) dan Mn(II) dari larutan air dilakukan pada suspensi lempung Bontang pada konsentrasi 2%. Larutan Cu(II) berair 2% dituangkan ke dalam suspensi tanah liat Bontang dengan konsentrasi awal 10, 50, 100, 250, 500, 1000, 2500 dan 5000 mg/dm3.

Aktivasi Adsorben

Kemampuan adsorpsi suatu adsorben dapat ditingkatkan melalui aktivasi fisik. Kenaikan diukur. Bahan halus tersebut dimasukkan ke dalam cold muffle furnace (Thermoyne type 48000) dengan cara oksidasi pada suhu sekitar 6000-7000C selama 2 jam (Jabłonska and Siedlecka, 2015; Alaistara dkk, 2018). Tungku kemudian dimatikan dan bahan aktif dibiarkan di dalam sampai suhunya menjadi dingin. Proses adsorpsi yang sama juga dilakukan pada tanah liat Bontang mentah.

Applikasi Adsorpsi

Kajian adsorpsi lempung Bontang pada logam berat dilakukan pada larutan sintetik yang dibuat berdasarkan senyawa murni analitik tembaga dan timbal yang mengandung salah satu ion logam tersebut yang konsentrasi awalnya mirip dengan konsentrasinya pada air asam tambang.

Penelitian ini dilakukan pada suspensi lempung dengan konsentrasi 2%. Suspensi dituang dengan larutan logam berat konsentrasi awal 10, 50, 100, 250, 500, 1000, 2500 dan 5000 mg/dm3. Sampel kemudian diaduk dengan vibrator putar menggunakan hot plate + pengaduk (Thermolyne type 1000 Stirrer plate) selama kurang lebih dua jam pada suhu 20oC kemudian ditempatkan di ruang gelap selama 22 jam. Selanjutnya, larutan dituangkan dan sedimen dihilangkan dengan clarifier sentrifugal pada 2500 rpm.

Massa logam yang terserap per satuan massa sedimen, q, dihitung dengan rumus sebagai berikut (Jabłonska dan Siedlecka, 2015).

$$q \text{ ¼ } \frac{C_i - C_e}{m_e} V \quad \frac{\Sigma_{mg}\Sigma^3}{mg} \qquad (1)$$

Catatan: Ci, Ce e konsentrasi awal dan kesetimbangan Cu(II) (mg/dm3), V e volume larutan (dm3), me massa sedimen yang digunakan (g). Tiga pengukuran dilakukan untuk setiap jenis sampel dan nilai rata-rata diambil untuk perhitungan lebih lanjut.

Analisis Adsorsi

Adsorpsi Isotermik

Adsorpsi isotermik mencerminkan hubungan jumlah adsorbat yang dilepaskan dari fase cair dengan satuan massa adsorben pada suhu yang sama (Hasan dkk, 2019). Jumlah logam teradsorpsi ditemukan dan isoterm adsorpsi q f(Ce) untuk Cu dan Mn pada lempung mentah dan tanah liat yang dikalsinasi ditentukan. Isoterm adsorpsi Freundlich dan Langmuir digunakan dalam penelitian ini karena keduanya sangat umum digunakan dalam deskripsi kuantitatif adsorpsi. Mereka menggambarkan hubungan antara massa adsorbat per satuan massa adsorben, juga disebut sebagai kapasitas adsorpsi (q, mg/g), dan konsentrasi adsorbat dalam cairan dalam kesetimbangan (Ce, mg/dm3). Rumus isoterm adsorpsi Freundlich memiliki bentuk sebagai berikut:

$$q \frac{1}{4} K_F C^n \; \eth mg = g\flat; \qquad\qquad (2)$$

dimana KF dan n adalah konstanta empiris tergantung pada adsorben dan adsorbat pada temperatur tertentu.Koefisien KF diukur kapasitas adsorpsi. Parameter n, dimana $0 < n < 1$, menunjukkan intensitas adsorpsi. Kurva yang berkaitan dengan nilai n yang kecil tumbuh tiba-tiba untuk konsentrasi kecil dan perlahan untuk yang lebih besar, mencerminkan adsorpsi yang kira-kira sama untuk semua rentang konsentrasi yang diperiksa.

Rumus isoterm adsorpsi Langmuir adalah sebagai berikut:

$$1/Q \quad Q \frac{K_L C_e \; \eth mg = g\flat;}{1 \flat K_L C_e} \qquad\qquad (3)$$

dimana KL dan Q adalah konstanta untuk adsorbat dan adsorben pada temperatur tertentu. Koefisien Q (mg/g) adalah kapasitas adsorpsi, ketika q cenderung untuk Ce besar. Konstanta KL (dm3/mg), disebut sebagai konstanta adsorpsi Langmuir, mengungkapkan kekuatan interaksi adsorbat dan adsorben. Nilai KL yang lebih besar menghasilkan kurva yang tumbuh tajam untuk konsentrasi kecil dan sebagian besar konstan pada tingkat Q untuk yang lain. Oleh karena itu, semakin besar nilai KL semakin rendah konsentrasi pada saat adsorben jenuh.

Ketika persamaan Langmuir dibandingkan dengan persamaan Freundlich, baik Q maupun KF merupakan faktor s yang mencerminkan kapasitas adsorpsi, dan baik KL maupun n menggambarkan bentuk isoterm. Namun, isoterm Freundlich menunjukkan nilai q tak terbatas untuk Ce besar, sedangkan isoterm Langmuir lebih realistis dan memberikan q dibatasi oleh Q. Koefisien KF, n, Q, KL, KE dan qm biasanya ditentukan dari bentuk linier dari persamaan isoterm. Dengan mengambil logaritma dari kedua sisi persamaan Freundlich diperoleh

$$\log q \; \tfrac{1}{4} \; n \log c_e \; \flat \; \log k_f; \qquad\qquad (4)$$

dimana log q adalah fungsi linear dari log Ce dengan koefisien log KF dan n. Bentuk linier mengarahkan kita untuk menggunakan metode regresi linier. Isoterm adsorpsi Freundlich dan Langmuir menunjukkan nilai koefisien determinasi yang sama. Hal ini menunjukkan bahwa adsorpsi pada lempung Bontang bersifat campuran dan tidak seragam serta tidak spesifik, yang kemungkinan berasal dari nonhomogenitas lempung. Situs adsorpsi dapat sangat berbeda dalam energi, tergantung pada apakah mereka berada di tepi atau dalam posisi cacat.

HASIL DAN PEMBAHASAN
Identifikasi Adsorben
Karakterisasi FTIR

Spektrum FTIR lempung Bontang ditunjukkan pada Gambar 1. Pada pra dan pasca aktivasi, lempung Bontang mengalami perubahan pergeseran bilangan gelombang Si-O-Si / Al-O-Al (dari 1031 cm-1 menjadi 1033 cm-1) dan Al-OH (dari 912 cm-1 menjadi 916 cm-1).

Mengacu pada penelitian yang dilakukan oleh (Bwapwa dkk, 2018), kalsinasi membuat perubahan seperti yang digambarkan dengan peningkatan intensitas akibat penghilangan pengotor (Tabel 1).

Tabel 1. Adsorpsi FTIR lempung Bontang sebelum dan sesudah aktivasi

| No	Gugus Fungsi	Angka gelombang (cm^{-1})		
		Referensi	Pra-aktivasi	Pasca-aktifasi
1	Al-OH-Al	3616	3695; 3623	3699; 3625
2	-OH	3500-3400	3421	3426
3	H-O-H	1639	1628	1635
4	Si-O-Si / Al-O-Al	1039	1002	1012

| 5 | Al-OH | 920-690 | 920 | 928 |
| 6 | Si-O / Al-O | 470,6 | 455 | 455 |

Karakterisasi Difraktometri Sinar-X (XRD)

Pola XRD lempung Bontang ditunjukkan pada Gambar 1. Analisis XRD lempung Bontang sebelum dan sesudah aktivasi diperoleh adanya kandungan mineral seperti kaolinit (K), montmorillonit (M), ilit (I), dan kuarsa (Q), ditunjukkan dengan munculnya puncak difraksi pada 2θ. Pada lempung Bontang pra-aktif, kaolinit, montmorillonit, ilit dan kuarsa muncul pada 35,02° dan 32,09°; 5,34°, 19,85°, dan 61,65°; 27,70°; dan 67,29° masing-masing. Pada lempung Bontang aktif, lempung Bontang, kaolinit, 2montmorillonit, ilit dan kuarsa muncul pada 30,24°, 39,35°, 60,2°; 5,65°, 19,10°, dan 62,05°; 26,89°; dan 64,37° masing-masing. Difraktogram pra dan pasca aktivasi menunjukkan penurunan intensitas dan puncak baru. Penurunan intensitas tiap mineral menunjukkan perubahan struktur dari crustal menjadi amorphous.
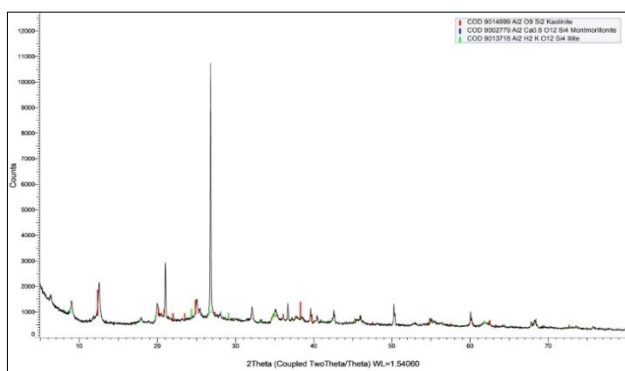
Gambar 1. Pola XRD lempung Bontang sebelum aktivasi
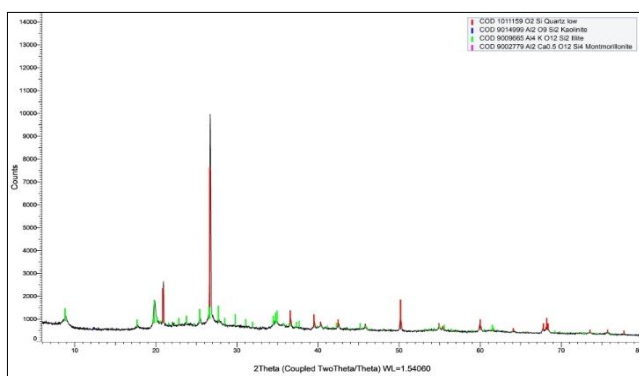
Gambar 2. Pola XRD lempung Bontang setelah aktivasi

Analisis Luas Permukaan

Angka luas permukaan merupakan interaksi antara suatu material padat dengan material di sekelilingnya, seperti cair dan gas. Hal terpenting dalam mendapatkan besaran luas permukaan adalah molekul gas terserap. Besaran luas permukaan ddidapatkan menggunakan uji adsorpsi-desorpsi N2 melalui persamaan Brunauer, Emmet, dan Teller (BET).

Pada penelitian ini, luas permukaan spesifik lempung Bontang pra-aktif sebesar 34,793 m2/g sedangkan lempung Bontang pasca aktifasi sebesar 49,859 m2/g. Pembesaran luas permukaan ini

kemungkinan disebabkan oleh proses aktifasi fisik. Luas permukaan yang diperbesar ini memberikan luas permukaan adsorben sehingga ion logam dapat teradsorpsi

AnalisisAdsorpsi

Tembaga

Adsorpsi statik pada lempung mengakibatkan perubahan konsentrasi Cu(II) yang ditampilkan pada Gambar 3
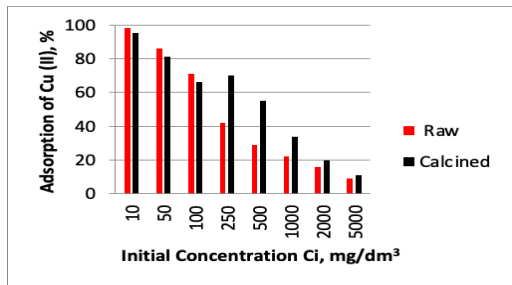
Gambar 3. Persentase Adsorpsi Cu(II) Pada Lempung Bontang

Terjadi penurunan persentase adsorpsi Cu(II) pada semua konsentrasi lebih besar seperti pada Gambar 4. Persentase adsorpsi maksimum lempung Bontang mentah pada Cu[II] adalah 96,2 % untuk konsentrasi awal 10 mg/dm3 dan 94,5% yang teraktivasi. . Turun menjadi 42,6% untuk 250 mg/dm3 dan 70,2% untuk yang dikalsinasi. Pada konsentrasi awal 5000 mg/dm3, itu sama dengan 8,3% untuk tanah liat Bontang mentah dan sekitar 7,2% untuk tanah liat aktif.

Lempung Bontang yang diuji memiliki kemampuan adsorpsi Cu(II) sama atau lebih baik dari adsorben lainnya.
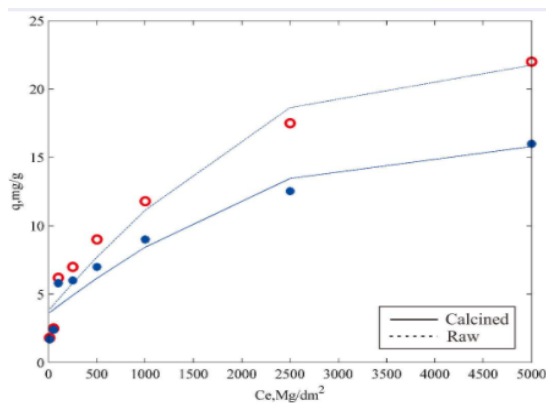
Gambar 4. Isoterm Adsorpsi Cu(II) Dari Lempung Bontang

Mangan

Persentase adsorpsi ion Mn tergantung pada konsentrasi awal. Semakin rendah dengan semakin tingginya konsentrasi awal Mn pada tanah liat Bontang yang diteliti.

Persentase adsorpsi ion Mn tergantung pada konsentrasi awal. Semakin rendah dengan semakin tingginya konsentrasi awal Mn pada tanah liat Bontang yang diteliti.

Seperti terlihat pada Gambar 5, untuk tanah liat Bontang mentah mengalami penurunan dari 74,62% untuk konsentrasi awal 10 mg/dm3 dan 76,67% untuk yang dikalsinasi. Pada konsentrasi awal 250 mg/dm3 mereka menurun menjadi 32,65% dan 44,29% masing-masing untuk sebelum dan sesudah kalsinasi. Konsentrasi awal 5000 mg/dm3, yaitu 4,33% untuk tanah liat Bontang mentah dan 6,87% untuk tanah liat aktif.

Gugus interpacket water dan OH— pada permukaan aluminosilikat sebagian besar dihilangkan dengan aktivasi lempung Bontang. Ini dibuat dalam mengurangi jumlah situs aktif bermuatan negatif pada permukaan logam.

Penurunan adsorpsi Mn(II) karena pelepasan gugus asam juga telah dipelajari dalam kasus karbon aktif yang diaktifkan pada suhu sekitar 600 ○C, meskipun karbon tetap berada di sebagian besar gugus non-asam permukaan.

Studi tersebut menunjukkan bahwa tanah liat Bontang yang diteliti dapat menyerap logam dalam jumlah besar.
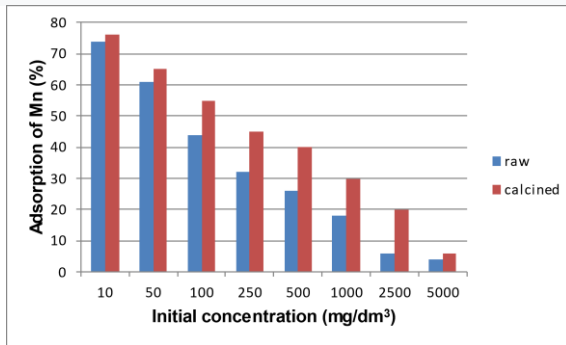
Gambar 5. Persentase Adsorpsi Mn(II) Pada Lempung Bontang
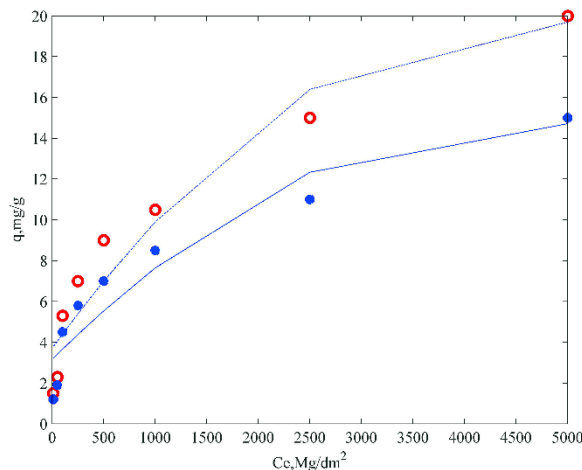
Gambar 6. Isoterm Adsorpsi Mn(II) Dari Lempung Bontang

Ringkasan Adsorpsi

Logam Cu[II] dan Mn[II] yang diadsorpsi oleh lempung Bontang yang diperiksa memenuhi kira-kira hubungan Cu(II) > Mn(II). Isoterm adsorpsi untuk Cu(II) dan Mn(II) (Gambar 5 dan 6) menunjukkan bahwa kapasitas adsorpsi tidak lebih dari 5 mg/g jika konsentrasinya kurang dari 100 mg/dm3.

Intensitas adsorpsi meningkat secara bertahap untuk Cu(II) dan Mn(II) ketika konsentrasinya lebih dari 250 mg/dm3. Adsorpsi Mn[II] yang lemah pada lempung Bontang dapat dikaitkan dengan adanya sejumlah kecil pirit halus pada sampel mentah, tetapi tidak ada pada pola XRD. Kehadiran oksidasi pirit dalam suspensi tanah liat dihidrolisis memimpin dalam pendudukan situs pertukaran dalam mineral tanah liat oleh ion hidrogen, yang bersaing dengan ion logam, secara signifikan mengurangi intensitas adsorpsi.

Tanah liat Bontang teraktivasi menunjukkan kemampuan adsorpsi yang sangat baik Mn[II]. Namun, kalsinasi tidak membuat perubahan signifikan pada struktur kristal untuk adsorpsi Cu[II]. Isoterm adsorpsi mencerminkan bahwa ion logam tidak membuat adsorben jenuh untuk konsentrasi yang relatif tinggi.

Hasil penelitian menunjukkan bahwa efisiensi pelepasan kation logam berat pada tanah liat Bontang mentah dan terkalsinasi tergantung pada struktur tanah liat dan bervariasi untuk masing-masing logam.

KESIMPULAN

Hasil analisis yang diambil mengarah pada kesimpulan berikut:

1. Tanah liat Bontang dapat dimanfaatkan sebagai material adsorben Cu(II) dan Mn[II] pada air asam tambang . Hal tersebut telah dibuktikan dengan penelitian ini, dimana diperoleh karakterisasi adsorben dan persentase adsorpsi.
2. Kalsinasi lempung Bontang pada suhu 600-700○C secara signifikan meningkatkan kemampuan adsorpsi Cu(II) dan penurunan Mn[II].
3. Kemampuan adsorpsi lempung Bontang terhadap ion tembaga lebih baik dibandingkan dengan kemampuan adsorpsi pada ion Mangan.
4. Persentase adsorpsi Cu(II) dan Mn[II] oleh larutan lempung Bontang berair meningkat dengan penurunan konsentrasi.
6. Pada penelitian ini adsorpsi tembaga terbaik adalah lempung Bontang teraktivasi sebesar 5.000 mg/dm3.

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