APPLICATION OF FA-GGBS GEOPOLYMER AS AN ALTERNATIVE ADSORBENT FOR HEAVY METALS REMOVAL

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by

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LIST OF ABBREVIATION

| FA | Fly Ash |
|---------|---|
| ICP-OES | Induction Couple Plasma – Optical Emission Sensor |
| GGBS | Ground Granular Blast-furnace Slag |
| LOD | Limit of detection |
| N/A | Not applicable |
| OFAT | One Factor at a Time |
| ppm | Part per million |
| rpm | Rotation per minute |
| XRD | X-ray Diffraction |
| XRF | X-ray Fluorescent |
| WHO | World Health Organization |

ABSTRAK

Sejak awal kewujudan, manusia menunjukkan kebergantungan yang kuat kepada sumber air terutamanya air permukaan. Walaubagaimanapun, semakin banyak sumber air permukaan dicemari oleh hasil sisa aktiviti manusia menjadikan sumber air berkurangan. Kepekatan logam berat di dalam sumber air permukaan menjadi isu yang sering terjadi. Jadi, kajian ini bertujuan 1) Menentukan komposisi elemen didalam penjerap dan larut-lesapan element oleh bahan penjerap, 2) mengkaji kemampuan GGBS geopolimer sebagai penjerap tembaga dan 3) menilai isoterma penjerapan menggunakan dua model iaitu Langmuir dan Freundlich berdasarkan data dari eksperimen. Keputusan menunjukkan bahawa kecekapan penjerapan tembaga adalah hampir 100% pada 1 g bahan penjerap, 150 rpm kadar putaran dan 3 jam proses penjerapan. Keputusan XRF menunjukkan GGBS geopolimer terdiri dari 3 elemen utama iaitu Silika, Kalsium dan Oksigen dengan masing-masing mempunyai peratusan komposisi 30.35%, 24.33% dan 18.26%. Berdasarkan data eksperimen, penjerapan yang terjadi mematuhi model isoterma Freundlich yang mengandaikan bahawa fenomena penjerapan yang terjadi hanya membentuk lebih dari satu lapisan diatas permukaan penjerap yang bersifat heterogen. Langmuir model juga menekankan bahawa hanya ada tindakbalas penjerapan antara bahan penjerap dan bahan pencemar, dan adat tindakbalas antara sesama bahan pencemar terjadi.

ABSTRACT

Since the beginning of time, humans have relied on surface water as a source of water. However, as more and more surface water are polluted by waste from human activities, this water supply is becoming less available. Elevated heavy metals concentrations in the surface water are one of the common issues. Adsorption has been proved to be the best process of water treatment because of its significant advantages. The adsorption assessment of GGBS shows good efficiency in copper removal as studied. GGBS is a by-product of the steel industry that has good structural and durable properties with fewer environmental effects. Therefore, this study aims to 1) To characterize the adsorbent based on the chemical composition using XRD, XRF, and leaching tests.; 2) determine the effectiveness of GGBS as an adsorbent in the removal of copper; 3) To compute adsorption isotherm analysis with Langmuir model and Freundlich model. Results showed very high efficiency of copper removal almost 100% with 2.5 g of adsorbent, 150 rpm as an agitation rate, and 3 hours as contact time. XRD study for GGBS showed high results for quartz (SiO₂), calcite (CaCO₃), and anorthite (CaAl₂Si₂O₈). While XRF study showed high results for elements Si, Ca, and O with 30.35%, 24.33%, and 18.261%, respectively. Based on the experimental data, the adsorption phenomena in this study follow the Freundlich Isotherm model which assumes that their adsorption occurred in multilayer order onto the heterogenous surface of the adsorbent. The Freundlich adsorption also expressing that there is possibility lateral interaction between adsorbates.

CHAPTER 1

INTRODUCTION

1.1 Introduction

Surface water has been a source of water supply since the existence of human beings. However, this water source is less abundant as before as more and more surface water are contaminated by waste from anthropogenic activity. The exploration of heavy metal's removal via adsorption approach has been received more attraction as many new materials are tested for their efficiency. Activated carbon, clays, and mineral rocks are the most popular adsorbent experimented with while researchers have actively increased their efficiency with chemical or heat treatment. Other than that, composite adsorbents are produced by merging two or more adsorbents physically by a platform such as alginate. Additionally, the incorporation between two adsorbents also taking part in the race of finding a better adsorbent material.

Geopolymers are inorganic polymer materials derived from alkali activation of aluminosilicate materials such as metakaolin, fly ash, and granulated blast furnace slag (GBFS) (Siyal et al., 2018). The geopolymer materials are regarded as environmentally friendly due to their low temperature of manufacturing (<100 °C) and lower CO_2 emission compared to standard cement (Maleki et al., 2019). The application of geopolymer can be found in various fields, and recently, scientists have experimented with it as an adsorbent due to its porous structure and low cost (Javadian et al., 2015).

Ground Granular Blast-furnace Slag (GGBS) and Fly Ash (FA) are selected as the material in the formation of GGBS/FA-based geopolymer. This material will be tested for its capability in immobilization of heavy metal that was selected which is copper.

1.2 Problem Statement

A high concentration of heavy metals in water can be fatal to humans and other organisms. Elevated concentration of heavy metal may directly affect humans via water consumption, as well as indirectly influence human health through food consumption that contaminated from the polluted water sources. The pollution of water due to the release of heavy metals into ecosystem metals has been causing worldwide concern. The main sources of heavy metals are the wastewaters from modern chemical industries such as metal plating facilities, battery manufacturing, fertilizer, mining, paper, and pesticides, metallurgical, mining, fossil fuel, tannery, and production of different plastics such as polyvinyl chloride (Ihsanullah et al., 2016).

This study is important as it is involving the exploration of new material as an adsorbent in the removal of unwanted and toxic metals. Copper for example has a number of applications in industrial and agricultural processes. Copper can be released into the environment from many sources. Drinking water can be a potential source for an intense copper exposition. Copper is highly toxic for drinking water, and mercury is the only metal more toxic than copper. Although copper is important for animal metabolism. However, the excessive ingestion of copper brings about serious threats, such as increased blood pressure and respiratory rates damaged in the kidney and liver, convulsions, cramps, vomiting, or even death (Ihsanullah et al., 2016).

Therefore, it is essential to immobilize the copper before it is contaminating the water source. the treatment process should be executed before the effluent release so that the risk of overexposure can be terminated. which will be done in this study by using GGBS as an adsorbent for cooper.

1.3 Objectives

This study aims to remove heavy metals from water, with an emphasis on copper, and to evaluate GGBS as an adsorbent for heavy metal removal.

Therefore, this study contains 3 main objectives which are listed below

- To characterize the adsorbent based on the chemical composition using XRD, XRF, and leaching test.
- ii. To determine the effectiveness of GGBS as an adsorbent in the removal of copper and the characterization of the geopolymers.
- iii. To calculate adsorption isotherm analysis with Langmuir model and Freundlich model.

1.4 Scope of Study

The capability of GBBS-based geopolymer as adsorbent is assessed by series of experiments. The contaminant concentrations are determined from the data gathered from previous study.

Three factors will be assessed including the initial concentration, contact time and adsorbent dosage. An OFAT (One Factor at a Time) approach will be implemented to acquire the optimum setting adsorption process.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Malaysia has been relying on surface water for the supply of fresh water for human consumption, agricultural and industrial use. However, more and more rivers have lost the 'clean' status day by day. In 2016, Natural Resources and Environment Minister had announced that 9% out of 473 rivers that have been monitored is considered polluted while 39% (slightly polluted) and 52% were clean (Figure 2.1) (Bernama, 2016). Although Malaysia has a plentiful amount of surface water and is replenished by a large volume of rainfall each year, the tremendous increase in population, along with the rapid expansion of industry and agriculture, places enormous strain on the present water supply. Climate change and a lack of clean water sources placed even more strain on the water supply.



Figure 2.1: Percentage of river status in 2016

Based on (WHO, 2017b), the source of water contamination can be divided into a few categories which are from a natural source, industrial and human dwelling, agricultural, water treatment setup, and pesticide as shown in Table 2.1. Natural sources include rocks, soils, and the impacts of geological setting and climate, while the rest may be ascribed to human activities, which account for the majority.

| Source of chemical constituents | Examples of sources Naturally |
|---|--|
| Naturally occurring | Rocks, soils and the effects of the geological setting and climate; eutrophic water bodies (also influenced by sewage inputs and agricultural runoff) |
| Industrial sources and human dwellings | Mining (extractive industries) and manufacturing and processing industries, sewage (including a number of contaminants of emerging concern), solid wastes, urban runoff, fuel leakages |
| Agricultural activities | Manures, fertilizers, intensive animal practices and pesticides |
| Water treatment or materials in contact with drinking-water | Coagulants, DBPs, piping materials |
| Pesticides used in water for public health | Larvicides used in the control of insect vectors of disease |

Table 2.1: Categorization of source of chemical constituents (WHO, 2017b)

The source of pollutants is also studied by Kamaruddin et al., (2015) concluded that the main pollution sources for the Terengganu River in Malaysia are municipal waste, surface runoff, agriculture runoff, organic pollution, and urban storm runoff, which are all anthropogenic sources. Chan et al., (2007) have listed the source of river pollutant in Malaysia which are agriculture (fertilizers, pesticides, and sediments from soil erosion), livestock farming (animal wastes), domestic homes (human wastes), urban areas (greywaters and untreated wastewaters) and industries (industrial effluent), but the most severe pollution comes from industrial effluent that discharged hazardous heavy metals into the river. Both studies mentioned above Chan et al., (2007) and Kamaruddin et al., (2015) have listed anthropogenic sources as the main pollutant in water bodies in Malaysia and the manufacturing industry has become the major contributor for hazardous chemicals including heavy metals.

2.2 Heavy Metals in Water Bodies

Heavy metals can be defined as the elements that possessed atomic weight from 63.5 to 200.6 with a specific gravity value of more than 5.0 and most of them are lethal to humans and other organisms (Sapingi, 2017). Heavy metals are not biodegradable, persistent, highly toxic and some are also carcinogens (Abdel-Shafy et al., 2019). As mentioned in Table 2.1, this pollutant may source from various origins.

As for effluent, there are two well established standards which are standard A and standard B (P.U. (A) 434., 2009). Standard A applies when the effluent is released in the catchment area located in the upstream of surface or above subsurface water supply intakes, for the purpose of human consumption including drinking water while for Standard B is applied for the location other than listed in Standard A. Copper's concentration permissible limit values for drinking water are 1 mg/L established by Malaysia authority and 2 mg/L by WHO. As for raw water Malaysia Authority has given similar value as it is permitted in drinking water. This is due to copper is irremovable via conventional method solely (WHO, 2017a). The value of maximum allowable limit for effluent for Standard A is 0.20 mg/L and standard B, 1.0 mg/L (Table 2.2).

Table 2.2: Maximum acceptable concentrations for copper (Ministry of Health (MOH), 2019; P.U. (A) 434., 2009; WHO, 2017a)

| Heavy | Drinkin | g water | Raw water | Standard A | Standard D |
|--------|----------|---------|-----------|------------|------------|
| metals | Malaysia | WHO | Malaysia | Stanuaru A | Standard D |
| Copper | 1 | 2 | 1 | 0.20 | 1.0 |

2.2.1 Copper in Surface Water

In a clean surface water source, the concentration of heavy metals less than permissible limit as most the metals are originated from natural sources such as dissolution of minerals. However, human activities surrounding this water source altered these attributes via agricultural, domestic, and industrial activities. Table 2.3 shows the summarized copper concentration in surface water in previous study which mostly evaluated river water. Studies by Kusin et al., (2017), Mandour, (2012) and Alsaffar et al., (2016) shows that the copper concentration is lower that acceptable limit for raw water as shown in Table 2.2. In Nile River, agricultural drainage water, sewage effluents and industrial wastes discharged into the canal, are the main reasons for the elevated concentration of heavy metal (Goher et al., 2014).

According to Arbabi and Golshani, (2016), there are many methods to remove the copper such as chemical precipitation, ion exchange, adsorption, and membrane filtration. Adsorption, the technique used in this study, is a well-known method for removing heavy metals in low concentrations from wastewater containing heavy metals. To remove heavy metal ions, a wide range of low-cost adsorbents have been designed and evaluated. However, the adsorption effectiveness is affected by the type of adsorbents used. Biosorption of heavy metals from aqueous solutions is a relatively recent technique that has shown great promise for heavy metal removal from wastewater (Arbabi and Golshani, 2016).

2.2.2 Copper Concentrations in Wastewater

Heavy metal-contaminated wastewater from mining, mineral processing, and a variety of manufacturing industries is one of the most difficult water pollutions to address, and copper is a common component of these effluents (Hu et al., 2017). The copper (Cu(II)) ion is vital for the development of enzyme synthesis, tissues, and bones in humans; nevertheless, too much Cu(II) can hurt the eyes and liver, as well as produce cellular process imbalances that lead to Menkes, Wilson, Alzheimer's, Parkinson's, and prion disorders (Hu, Li et al. 2017).

In Table 2.3, the highest concentration of copper can be seen in mixed Industrial wastewater from plating plant. as high as 156 mg/L (Lee et al., 2017). A high concentration of copper also founded in the untreated plating effluent sample with a maximum concentration of 152.46 mg/L (Qin et al., 2018a).

| Categories | Location/Type | Copper | Reference |
|---------------|----------------------|--|-----------------------------|
| Surface water | Selangor, Malaysia | 0.00-0.05 | (Daniel and Kawasaki, 2016) |
| | Pahang, Malaysia | <lod-0.01< td=""><td>(Kusin et al., 2017)</td></lod-0.01<> | (Kusin et al., 2017) |
| | Penang, Malaysia | 0.004-0.006 | (M. Alsaffar et al., 2016) |
| | Perak, Malaysia | 0.005-0.019 | (Akinbile et al., 2013) |
| | Nile River, Egypt | 0.003-0.021 | (Goher et al., 2014) |
| | Dakahlyia, Egypt | 0 | (Mandour, 2012) |
| Wastewater | POME | n/a | (Shavandi et al., 2012) |
| | Industrial (mix) | 0.29 +0.06 | (Hegazy et al., 2011) |
| | Industrial (mix) | n/a | (Ibrahim and Elsayed, 2019) |
| | Paper Mill (treated) | < 0.01 | (El-Awady et al., 2019) |
| | Plating (treated) | n/a | (Oden and Sari-Erkan, 2018) |
| | Plating | 75 ± 5 | (Ilhan et al., 2019) |
| | Plating | 152.46 | (Qin et al., 2018b) |
| | Plating | n/a | (Zhou et al., 2020) |
| | Paper mill (treated) | n.d | (Hanafiah et al., 2020) |
| | Industrial (mix) | 35-156 | (Lee et al., 2017) |

Table 2.3: Copper's concentrations in previous studies

2.3 Treatment for Heavy Metals

There are many treatment approaches have been explored including phytoremediation, coagulation and flocculation, ion exchange, ultrafiltration, membrane separation, advance oxidation process and adsorption. The list of advantages and disadvantages of each approach itemized in Table 2.4. Because of its high efficiency, cost-effectiveness, and simplicity, adsorption is recommended as an effective and inexpensive approach for removing cooper ions from aqueous industrial wastes (Arbabi and Golshani, 2016).

| Treatment | Advantages | Disadvantages |
|----------------------|------------------------------|---------------------------------|
| Chemical | - Simple | - Large amounts of sludge |
| Precipitation | - Inexpensive | produced |
| | - Most of metals can be | - Disposal problems |
| | removed | |
| Chemical | - Sludge settling | - High cost |
| coagulation | - Dewatering | - Large consumption of |
| | | chemicals |
| Ion-exchange | - High regeneration of | - High cost |
| | materials | - Less number of metal ions |
| | - Metal selective | removed |
| Adsorption | - Most of metals can be | - Cost of adsorbent |
| | removed | - No regeneration |
| | - High efficiency (>99%) at | - Performance depends upon |
| | low concentration | adsorbent |
| | - Relatively less costly | - Low efficiency at high |
| | materials | concentration |
| | - Easy operating conditions | - Saturated adsorbent need to |
| | - large potential for ion | be disposed properly |
| | exchange | |
| Biological / | - Biologically degradable | - Requires additional nutrients |
| phytoremediation | - | - Technology yet to be |
| | | commercialized |
| Membrane process | - Less solid waste produced | - High initial and running cost |
| and ultra-filtration | - Less chemical consumption | - Low flow rates |
| | - High efficiency (>95% for | - Removal (%) decreases with |
| | single metal) | the presence of other metals |
| | | - High energy consumption |
| Advance oxidation | - Rapid reaction | - Less effective |
| Process | - Easy to add as enhancement | - Toxic residual of oxidation |
| | for existing treatment plant | agent |

Table 2.4: The advantages and disadvantages of heavy metal removal treatment (Acharya et al., 2018; Bhatnagar and Sillanpaa, 2017; Uddin, 2017)

2.4 Adsorption

Based on Arbabi and Golshani, (2016) & Hari, (2017), adsorption has been proved to be the best process of water treatment because of its significant advantages. Adsorption process is a suitable technique for inorganic and organic pollutants removal from wastewater, because of the significant advantages like low-cost, availability, profitability, ease of operation, efficiency, and effectiveness than other techniques (Uddin, 2017). The process involves separation of a substance from one phase and its accumulation at another surface.

2.5 Ground Granulated Blast-furnace Slag (GGBS)

GGBS is a by-product from steel industry which has good structural and durable properties with less environmental effects (Saranya et al., 2018). The adsorption assessment of GGBS shows good efficiency in copper removal as studied by Hari, (2017) where the removal efficiency recorded as high as 96.5%. In a form of geopolymer, Maleki et al., (2020) found that the GGBS geopolymer has the capability to adsorb methylene blue dyes up to 80%.

Nonetheless, the number of study for GGBS/FA-based geopolymer is still low, thus this study will help to expand the knowledge of usability of GGBS in water treatment generally, particularly in heavy metal removal.



Figure 2.2: GGBS

2.6 Adsorption Isotherm

Langmuir isotherms model was established with three major assumptions which are the adsorption only occurred in monolayer onto the surface, the surface is homogenous (the availability of adsorption site is distributed fairly on the adsorbent surface, and the is no lateral interaction between adsorbate (Bergaoui et al., 2018). The illustration comparison between adsorbate- adsorbent interaction and adsorbate lateral interaction as depicted in Figure 2.3

The general equation for Langmuir isotherms is as below as in Equation 2.1

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$$
 Equation 2.1

Where q_e is the adsorbed amount at equilibrium, C_e is the equilibrium contaminant concentration, K_L is the Langmuir equilibrium constant, and q_m is the maximum adsorption capacity.

The constant in the Langmuir isotherm formula can be obtained by linearization of the formula above. There are 4 types of linear formulas derived from the main equation (refer to Table 2.5). Based on the slope and the y-intercept computed in the linear regression of each type of equation, the constant value of q_m and K_L can be obtained.

| Туре | Equation | |
|------|--|--------------|
| 1 | $\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \left(\frac{1}{q_m}\right) C_e$ | Equation 2.2 |
| 2 | $\frac{1}{q_e} = \left(\frac{1}{K_L q_m}\right) \frac{1}{C_e} + \frac{1}{q_m}$ | Equation 2.3 |
| 3 | $q_e = q_m - \Bigl(rac{1}{K_L}\Bigr)\Bigl(rac{q_e}{C_e}\Bigr)$ | Equation 2.4 |
| 4 | $\frac{q_e}{C_e} = K_L q_m - K_L q_e$ | Equation 2.5 |

Table 2.5: Linear equation derived from Langmuir Isotherm equation (Guo and Wang, 2019)



Figure 2.3: Illustration of Normal adsorption interaction and Lateral Interaction

Additional constant value (R_L) can be derived from Equation 2.6. The value of R_L represented the feasibility of the adsorption occurred following Langmuir isotherm model.

$$R_L = \frac{1}{(1 + K_L C_0)}$$
 Equation 2.6

Where R_L is the Separation Factor, K_L is Langmuir equilibrium constant, and C_0 is the initial concentration of the contaminant. The value of R_L and what it signifies is summarized in Table 2.6. The value is important to validate the agreement between the experimental value with the model

| Value of R _L | Description |
|-------------------------|--------------|
| $R_L > 1$ | Unfavorable |
| $0 < R_L < 1$ | Favourable |
| $R_L = 1$ | Linear |
| $R_L = 0$ | Irreversible |

Table 2.6: The description of R_L value (Ayawei et al., 2017)

Conversely, Freundlich Isotherm considers the heterogeneity surface of an adsorbent surface. This assumption describes that the adsorption occurred onto the surface with different adsorption energy. It is also expressing the adsorption that occurred in a monolayer composition. The general equation for Freundlich as Equation 2.7 below.

$$q_e = K_f C_e^{\frac{1}{n}}$$
 Equation 2.7

Where q_e is the adsorbed amount at equilibrium, K_f is the Freundlich constant, C_e is the concentration of contaminant at equilibrium and n is heterogeneity factor.

Similar to the Langmuir Isotherm, the equation is linearized to obtain the constant that listed previously. The linear equation as below (Equation 2.8).

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \qquad \qquad \text{Equation 2.8}$$

Where 1/n values signify the type of isotherm whether it is irreversible (1/n = 0), favourable (0 < 1/n < 1) and unfavorable (1/n > 1). *n* value describes the heterogeneity of the adsorption surface; a smaller 1/n value indicates a more heterogeneous surface and an n value between 1 and 10 indicates a favorable process (Edet and Ifelebuegu, 2020).

CHAPTER 3

METHODOLOGY

3.1 Introduction

In this section, the method and the approach used in the objectives listed previously in Chapter 1 will be thoroughly discussed. Based on objectives, there are three parts, and the overall flowchart of this study is depicted in Figure 3.1.

In the first part, the focus will be on the data collection of the previous study. There are two groups of data is collected, which are the concentration of copper in surface water and industrial wastewater. The data collected will be used in the determining initial concentration of copper in synthetic water.

The adsorbent used in this study is GGBS –based on geopolymer where its character such as elements and minerals content will be revealed. The most important part of this study is the adsorption experiment where three factors are investigated. The factors include agitation rate, adsorbent dosage, and contact time. The last part of this study is involving isotherm models derivation using experimental data. Two types of isotherms models are used in this study.



Figure 3.1: Overall flowchart of study

3.2 Synthetic Sample Preparation

The application of synthetic solution in water treatment research has been a widely selected approach by researchers (Al-Harahsheh et al., 2015; Nasr et al., 2020). This is due to less disturbance from unknown compounds and manageable initial concentration. These factors help to generate more explainable results as well as good in repeatability and reproducibility.

Copper concentrations in prior research are shown in Table 3.1 and it was used to set the initial concentration in this study. However, a concentration of 12 ppm was chosen for this study. A copper 1000 ppm stock solution was diluted to 12 ppm by adding it to ultrapure water based on the dilution Equation 3.1:

$$M_1V_1 = M_2V_2$$
 Equation 3.1

Where M₁ - Initial concentration

- V_2 Volume of solution
- M_2 The concentration needed
- V₂ Volume of solution needed

2L of copper solution with 12 ppm concentration was prepared and kept in a cold room to be used later.

| Reference | (Cu) Concentration | (Cu) Type |
|--|-----------------------|---|
| (Hari, 2017) | 10 to 100 mg/L. | Copper sulphate pentahydrate |
| (Kim et al., 2008) | 10 - 5000 mg/L | Copper sulphate pentahydrate |
| (Aydin et al., 2008) | 25-500 mg/L | Copper(II) nitrate (Cu(NO ₃) ₂ · 3H ₂ O) |
| (Feng et al., 2009) | 50 and 100 mg/L | Copper sulphate pentahydrate |
| (Veli and Alyüz, 2007) | 20,100,160 mg/L | Copper(II) nitrate (Cu(NO ₃) ₂ · 3H ₂ O) |
| (Netzer and Hughes, 1984) | 10 mg/L | Copper sulphate pentahydrate |
| (Liu and Zhou, 2010) | 50 mg/L | Copper sulphate pentahydrate |
| (Periasamy and Namasivayam, 1996) | 10 to 25 mg/L | Copper sulphate pentahydrate |
| (Viraraghavan and Dronamraju, 1993) | 1 mg/L | Copper sulphate pentahydrate |
| (Özer et al., 2004) | 100 mg/L | Copper(II) nitrate |

Table 3.1: Copper concentrations in prior research

3.3 Adsorption Study

In this section, the adsorption study will be discussed in details. An OFAT approach was used to run the experiment where three factors are involved.

3.3.1 Filtration

A vacuum filtration setup (Figure 3.2) was used to filter the samples before sending them to ICP-OES. To extract just the soluble part of the metal, a 0.45 μ m pore size membrane filter (Figure 3.3) was utilized. After the membrane filter and vacuum tube were installed, a strong clamp was placed to hold the funnel. The samples were then placed into the funnel, and the air compressor was turned on. The liquid in the filter flask was utilized to collect samples for ICP-OES.



Figure 3.2: Vacuum filtration setup



Figure 3.3: Membrane filter

3.3.2 Metal concentrations using ICP-OES

During the characterization and experimental phases, ICP-OES (Figure 3.4) was utilized to measure the concentration of each heavy metal. Because of its ability to produce a multi-metal concentration in one run, ICP-OES was chosen over AAS. ICP-OES is a piece of equipment that causes the elements to be excited into an unstable state known as plasma. To return to a stable state, the exciting components remove surplus energy by radiating electromagnetic waves with distinct wavelengths. Each element generates a distinctive wavelength wave, and the intensity of each wavelength reflects the element's concentration. A sensor inside the apparatus detects the intensity of the electromagnetic wave that was emitted.10 ml of samples were placed into an ICP tube, and the sample was preserved with HNO₃.



Figure 3.4: ICP-OES

3.3.3 Leaching Study

A leaching study was done to determine the soluble metal originated from the adsorbent and to know which metal has a positive correlation with the dosage of adsorbent. The test was set up with six 250 mL conical flasks, each filled with 100 mL

ultrapure water. With a 0.5 g interval and one control run, the dosage ranged from 0 to 2.5 g. The contact time and rotation speed were set to 2 hours and 150 rpm, respectively. ICP-OES was used to determine the concentration of heavy metals.

3.3.4 Effect of Agitation Rate

For this test, six conical flasks were used to evaluate the effect of varied speed variations. Each flask contains 100 mL of ultrapure water, and the amount of adsorbent and contact time were set to 1 g and 3 hours, respectively. The rotation speed ranged from 50 to 300 revolutions per minute, with 50-rpm gaps between each set. ICP-OES was used to measure heavy metal concentrations. Three arrangements were carried out, with the average data being presented.

3.3.5 Effect of Adsorbent Dosage

The test included 6 sets of 250 ml conical flasks, each filled with 100 ml of ultrapure water, similar to the agitation rate experiment. With a 0.5 g interval and one control run, the dosage ranged from 1 g to 3.5 g. 3 hours and 150 rpm were chosen as the contact time and rotation speed, respectively. ICP-OES was used to determine the concentration of heavy metals. Three arrangements were carried out, with the average data being presented.

3.3.6 Effect of Contact Time

There was one control run and contact time settings ranging from 15 minutes to 2 hours. The first two sets of studies had 15-minute intervals (between 15 and 30 minutes), whereas the following two sets had 1 hour and 2 hours intervals. The dosage and rotation speed were set to 1 g and 150 rpm, respectively. Three arrangements were carried out, with the average data being presented.

3.3.7 Adsorption Isotherm

Two isotherm models were selected in this study which is Langmuir Isotherm and Freundlich Isotherm. The models are computed by employing the experimental result. As explained in Chapter 2, a linear model for each type of isotherm was computed and the constant values are calculated from the slope and intercept values established from the linear equation. A comparison of these values comprehends the suitability of an isotherm model with the experimental data. All the plots and regressions were determined using OriginPro 9.0.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Adsorbent characterization

GGBS is a by-product of the steel industry that has good structural and durable properties with fewer environmental effects. To characterize the adsorbent based on the chemical composition the tests XRD, XRF, and leaching were conducted. Each of these tests result is supporting others.

4.1.1 Chemical composition by X-ray diffraction (XRD)

Ground granulated blast furnace slag and commercial Class F fly ash were utilized as solid predecessors in this study. X-ray fluorescence was used to examine their primary chemical components. Figure 4.1 shows the X-ray diffraction patterns of the utilized slag and fly ash.



Figure 4.1: XRD result for GGBS based Geopolymer produced