CHAPTER THREE

MATERIAL AND METHOD

The use of natural waste materials to treat wastewater containing heavy metals because its high adsorption potential and also it is a cheap source to prepare low cost adsorbent. In this chapter, we will try to description of the adsorbent, chemicals and adsorbate that were used throughout the study and also the regeneration methods, isotherm procedure as well as analytical methods. Also the characterization of the adsorbent as its use for the removal lead.

3.1 Cleaning of glassware

The glassware which used in the experiments were first washed with detergent and properly rinsed with tap water, this was followed by the addition of 10% nitric acid to further rinse and get remove any residual metal. The glassware was then finally rinsed with tap water and then distilled deionized water. The cleaned glassware was dried prior to use in experiments.

3.2 Raw material and chemicals

Raw jatropha seed husk was collected from a jatropha seed mill. Initially, the raw jatropha seed husk was cleaned, dried, blended and sieved into sizes of 250 μm and subjected to chemical modification where the heavy metal salts used in this study. $H_2SO_4(70\%)$ was used for jatropha seed husk chemical modification. Also the HCl (37%) and NaOH were used to adjust the pH of lead solutions.

3.3 Adsorbate

The adsorbate used in this study was lead. This element was chosen as adsorbate because it has the properties of an ideal adsorbate and it is also a priority pollutant.

3.4 Modification of jatropha seed husk:

The crushed jathropha seed husk will be collected from nearby oil mill. The adsorbent thus obtained was sieved through sieve on 250 µm. It will be washed with deionized water for several times to remove foreign particles and oven dried at 110 °C over night. 50 g of seed will be activated with 250 ml of concentrated sulfuric acid (70%) by heating the mixture for 6 hours at 100°C with a magnetic stirrer placed inside it. The slurry obtained was filtered by using whattman filter paper. The filter cake was repeatedly washed with deionized water and dried in an oven a 110 °C for 12 hours before use.

3.5 Preparation of lead solution:

Stock solution of Pb(II), about 1000 mg/l was prepared by transferring 1.626 g of lead(II) nitrate in a 1000-mL volumetric flask. Prior to dilution with deionized water to 1 L volume, a few drops of 0.1 M HNO_3 acid were added to dissolve the salt completely. Various concentrations of test solution ranging from 50 mg/l to 300 mg/l were prepared by subsequent dilution of the stock solution using deionized water while the initial pH was adjusted to 5.5±0.2. Fresh dilution of the stock solution was done for each sorption study.

3.6 Calibration Curve Solutions

10 mL of the stick solution was taken into a 100-mL volumetric flask to make lead solution concentration at about 100 mg/L. From this solution, 2mg/L, 4mg/L, 6 mg/L, 8 mg/L and 10 mg/L solution were prepared by using the following formula:

$$M_1V_1=M_2V_2 \qquad (3.1)$$

Where,

M_1 high or original concentration

V_1 volume needed of original concentration

M_2 low or new concentration

V_2 total volume of new concentration

3.7 Effects of initial concentration and contact time:

The adsorption of lead (II) ions on the dried jatropha seed husk was investigated in batch adsorption equilibrium experiments. The experiment was performed by mixing 0.1 g of dried jatropha seed husk in 50 mL of the synthetic metal solutions. The pH of solution was 5.5±2. The mixtures were mixed slowly with a rotary shaker at a rate of 150 rpm for 240 min at temperature room. The solid jatropha seed husk was separated from the liquid phase with membrane filter 0.45 μm, The concentration of lead (II) remaining in residual solution after a time lapse from the beginning of the experiment was determined by taking the residual solution absorbance on by ICP. The above was conducted on five different initial concentrations of lead (II) i.e., 50, 75, 100, 200 and 300 mg L^{-1}.

3.8 Equilibrium Uptake Experiment:

Each of the batch adsorption will be carried out by contacting the surfactant modified seed husk with the metal ions solution under different condition for a period of time in 100ml plastic container. The uptake capacity of Pb(II) will be studied in a systematic way in this work. Experiments will be conducted at (30 ±1) ° C temperature to study the effect of initial pH, contact time and initial metal ion concentration. Each experiment will be triplicated under identical condition. To minimize error and the mean value will be used for calculation.

To study the effect of temperature, each experiment will be carried out in a thermoregulated water bath (Haake Wia Model, Japan) with shaker cover in order to prevent heat loss to the surroundings and the residual metal ion concentrations will be analyzed by using Atomic Absorption Spectrophotometer (Shimadju AAS, Japan). The equilibrium adsorption amount (q_e) is calculated according to equation (1)

$$\mathbf{q_e = \{(C_0 - Ce)\}V/W} \qquad\qquad \mathbf{3.2}$$

where, Co is the initial metal concentrations in the solution (mg metal L^{-1} fluid), Ce is the metal concentrations in the solution (mg metal L^{-1} fluid) at time t, q_e is the adsorption amount of metal ion (mg/g) at equilibrium contact time, *W* is the weight of adsorbent (g), *V* is the volume of solution (ml).

3.9 Kinetic studies

The procedures for kinetic test are basically identical to those of equilibrium test. The aqueous solutions were sampled at various time intervals (5, 10, 15, 30, 60, 120 and 240 min) for Jatropha seed husk samples modified by $H_2SO_4.$ The concentration of lead were

similarly measured. The amount of adsorption at any time t, q_e (mg/g) was calculated by Equation:

$$q_t = (Co - Ct) \times V/W \qquad (3.3)$$

Where, qt the amount of lead adsorbed at time t(mg/g), Co and Ct(mg/l) are the liquid phase concentration of lead at initial and any time t, respectively, V is the volume of the solution (L) and W is the mass of adsorbent(g).

3.10 Data analysis

The amount of Pb^{2+} and adsorbed (a_t) in mg/L from lead solution by the adsorbent at the various time t was computed by using the following equation:

$$a_t = Co - Ct \qquad (3.4)$$

The percentage of Lead metal removed from the aqueous solutions by the adsorbent (% Rem) was computed using equation 2:

$$\% \ Rem = (Co - Ct) \times 100 / Co \qquad (3.5)$$

Where Co and Ct are the initial metal ion concentration and metal ion concentration at time, t respectively in mg/L.