Original Article

Synthesis and Characterisation of Metal-Organic Frameworks (MOFs) Using Benzene-1, 4- Dicarboxylic Acid as a Linker for Chromium (VI) Adsorption

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Abstract - Metal-organic frameworks (MOFs) applied in adsorption processes have shown appreciable effectiveness for removing dissolved ions. This study studied the adsorption rates of chromium (VI) ions by synthesised MOFs (metal-organic framework) using batch adsorption methods. The Na-MOF, Zn-MOF, and Mg-MOF were synthesised solvothermal from the reaction of sodium, zinc and magnesium nitrate salt with Benzene-1, 4- dicarboxylic acid, respectively, to yield the adsorbents. The synthesised adsorbents, Na-MOF, Zn-MOF, and Mg-MOF, were named RSU-1, RSU-2 & RSU-3, respectively. The results showed that the synthesised adsorbents exhibited good adsorption rates at distinct capacities. The BET values reflecting the surface areas of the synthesised adsorbents, RSU-1, RSU-2 and RSU-3, are 371.958 m²/g, 404.248 m²/g, and 547.372 m²/g, respectively. Adsorption rates were observed to increase as pH, concentration of chromium (VI) ion and contact time increased. The increase in adsorption as the pH tended to alkalinity (pH 2-pH 10) was from 2,240 mg/g to 19,580 mg/g for RSU-1, 1,550 mg/g to 14,110 mg/g for RSU-2, and 8,940 mg/g to 15,900 mg/g for RSU-3 adsorbents. Maximum Adsorption capacity was observed at pH 10, adsorbate concentration of 100 mg/l and contact time of 10,800sec. Increase in contact time from 3600-10,800sec increased adsorption rate from 3,340 mg/g to 7,242 mg/g for RSU-1; 6,155 mg/g to 9,972 mg/g for RSU-2; and 3,750 to 4,880 mg/g for RSU-3. As concentration increased from 10-100 mg/l, there was an increase in adsorption capacities from 923.7 mg/g to 10,121 mg/g for RSU-1, 1,650 mg/g to 10,570 mg/g for RSU-2, and 967.0 mg/g to 11,873 mg/g for RSU-3. The rate of percentage removal of the adsorbates was in the order of RSU-1> RSU-3> RSU-2. The order is attributed to the effect of the ionic size of the adsorbents. Isotherm data fitted suitably with the Freundlich model, thus suggesting good adsorption from the solution. The kinetic model was found to have fitted the pseudo-second-order model, suggesting chemisorption for the synthesised adsorbents. The metal-organic framework can be effectively employed as an adsorbent for an enhanced adsorption capacity of dissolved ions on the premises of its large surface area.

Keywords - Metal-Organic Frameworks, MOFs, Benzene-1, 4- Dicarboxylic Acid, Adsorption.

1. Introduction

Within the last two decades, the existence of Metal-Organic Frameworks (MOFs) has become a reality. It is also referred to as porous coordination polymers (PCPs) [1] or porous coordination networks (PCN). MOFs are a distinct crystalline material that contains a metal as a central ion surrounded by an organic ligand [2]. These advanced materials can be likened to sponges with special abilities to take up, hold and release molecules from their pores. MOFs are typically appreciated for their large surface area, tuneable pore size and various applications. In solvothermal synthesis, MOFs are produced through chemical reactions in non-aqueous solutions at high temperatures. It is known to produce MOFs with the best crystalline and morphological features [3].

Bridging ligands are important building units in the construction of MOFs.

Hence, to control the structure of a MOF material, the selection of rigid, organic linkers is one of the most crucial decisions [4]

Such ligands would be of considerable importance as precursors for the preparation of modern materials such as Metal-Organic Frameworks (MOFs) or Covalent Organic Frameworks (COFs), which have been reported in the literature to have a variety of potential applications such as gas storage, gas purification adsorption of heavy metals, catalysis, drug delivery, etc. [5].

The issue of heavy metal pollution is increasingly pervasive on a global scale. Heavy metals are naturally occurring elements that are present in the Earth's crust. However, excessive amounts of heavy metals can pose a significant risk. Their geochemical cycle and biochemical content have drastically altered due to man's indiscriminate activities [6].

Heavy metals are a natural makeup of the Earth's crust and have been a major pollutant of terrestrial and aquatic environments [7]. Heavy metals ions such as chromium, copper, lead, zinc, mercury, and cadmium are present in wastewater from various industries such as electroplating, tanning factories, steel works, wood preservation, plastic procession, metallurgical and mining activities, atomic power plants, dyes and pigments, chromate preparation, ceramic paints, and fertiliser industries [8].

According to the World Health Organization (WHO), long-term exposure to Cr (VI) levels of over 0.1 ppm causes respiratory problems, liver and kidney damage, and carcinogenicity. Therefore, it is necessary to treat effluents to reduce the Cr (VI) concentration in water and wastewater prior to their transport and cycling in the environment [WHO].

The rising concern is their toxicity, persistence in natural conditions, ease of incorporation into the food chain and web, and non-biodegradability. Also, the activities of man have in no mean amount contributed to the degradable and deplorable state of the environment.

In order to remove Cr (VI) from the aquatic environment due to its high level of toxicity and non-biodegradability [AlIan], a wide number of technologies have been adopted with the success of these procedures depending on Cr(VI) concentration and nature of Cr(VI) sources. Commonly used removal procedures are adsorption [9]. Adsorption is one of the most efficient ways to remove heavy metals from wastewater [10].

Because of this recent development of concern towards man and his environment, the need for an effective adsorbent that can enhance the removal of such pollutants is borne. To this end, we report herein a series of Metal-Organic Framework: RSU-1, RSU-2, RSU-3

2. Materials and Methodology

2.1. Synthesis of MOF Adsorbent

The synthetic method is shown in Scheme 1.



Scheme 1: Synthesised reaction between metal ions and an organic ligand.

Where X = Sodium nitrate NaNO₃, Zn (NO₃)₂ & Mg (NO₃)₂

The MOF adsorbents were synthesised using a mixture of dimethylacetamide (DMA)/water (1:1) and 1.25mmol (0.2075g) of benzene-1, 4-dicarboxylic acid (BDCA). 0.1063g of NaNO₃, 0.3550g of Zn (NO₃)₂, and 0.3210g of Mg (NO₃)₂ were mixed in water in a clean beaker and stirred for 600sec using a magnetic stirrer at 150°C, respectively. The organic ligand – Benzene-1, 4-dicarboxylic acid was equally mixed with the solvent –dimethylacetamide (DMA). The resulting solution was transferred into the bomb reactor plate and firmly sealed. The firmly sealed bomb reactor was put into an oven and heated at 150°C for 24hrs. The resulting product was allowed to cool slowly for 6 hours to obtain colourless block crystalline material at the bottom of the bombed reactor. The solvent was decanted, and the crystalline material was airdried.

The Synthesis of RSU-1, RSU-2, and RSU-3 follows a similar procedure. The synthesised adsorbents, Na-MOF, Zn-MOF, and Mg-MOF, were named RSU-1, RSU-2 & RSU-3, respectively.

2.2. Analysis and Characterisation of Adsorbent Surface Morphology

The surface morphology of the synthesised adsorbents was analysed at the Laboratory- Centre for Petroleum Geoscience, University of Port Harcourt, and Rivers State using the X-ray diffraction machine by Olympus. Powder Xray diffraction has proven to be an important tool in phase identification, sample purity, crystalline size and, most often, morphology [Cameron & Raymond 2019]. The properties of the synthesised adsorbents were also determined using Brunauer-Teller-Emmet (BET) for surface measurements at the Ahmadu Bello University Zaria by a Quanta chrome instrument (NOVA station A, version 11.03, USA) based on the principle of adsorption/desorption of nitrogen at 77k and 240/240s (ads/des) equilibrium time.

The Barrett-Joyner Halenda (BJH) was also employed to determine the pore area and specific pore volume using adsorption and desorption techniques. This technique characterises pore size distribution independent of the external area due to the particle size of the sample.

2.3. Preparation of Chromium (VI) Solutions

Stock solution (1000 ppm) of chromium (VI) ion was prepared by dissolving 2.828 g of potassium dichromate ($K_2Cr_2O_7$) in 1 litre of deionised water. The working solutions (10, 20, 40, 60, and 100 ppm) were prepared from the stock solution by appropriate dilution of the potassium dichromate ($K_2Cr_2O_7$) stock solution with deionised water.

The samples were analysed for chromium using the GBC Xplor AA atomic absorption spectrophotometer instrument (manufactured in Australia) as stated in the Operational Manual (GBC, 2016). Sets of known standards were used, and one of them was used for quality control.

2.4. Batch Adsorption Studies

Batch Adsorption Experiments were conducted using 75 mL beakers into which the chromium (VI) solution and the synthesised adsorbent were added. The beakers were agitated on the orbital shaker at a constant speed of 300 rpm.

The samples were withdrawn at a predetermined time and filtered using the Whatman No. 1 filter paper. The filtrates were analysed for residual chromium (VI) concentration using an atomic absorption spectrophotometer (AAS).

All pH measurements were made using the pH Meter (HANN instrument: H196107 pH).

All the experiments were carried out at room temperature. The percentage of chromium (VI) ion removed by the synthesised adsorbent was calculated from the differences between the initial (C_o) and final (C_f) chromium (VI) concentration using equation 1. The amount of chromium (VI) ion adsorbed (q_e) was calculated using equation 2.

% Removal=
$$\frac{\text{Co-Cf}}{\text{Co}} \ge 100$$
 (1)

$$q_e = \frac{Co - Cf}{m} \quad x \quad V \tag{2}$$

Where q_e is the amount of chromium (VI) ion adsorbed (mg/g), C_o is the initial concentration (mg/l), C_f is the final concentration after treatment (mg/l), V is the volume of solution (l), and m is the mass of the adsorbent (g).

2.5. Determination of the effect of pH on Adsorption of Cr (VI) ion

Precisely, 0.5 g of each of the synthesised adsorbents (RSU-1, RSU-2, and RSU-3) was weighed and added to a beaker containing 20 mL of 40 ppm chromium (VI) solution. The pH adjustment was actualised by adding HCl (0.1M) or NaOH (0.1M) solution drops.

The mixture was covered and equilibrated on an orbital shaker at 300 rpm for 60 minutes. The mixture was filtered, and the residual chromium (VI) concentrations were determined using AAS. The procedure was repeated at another pH (2.0, 5.0, 8.0, and 10.0) for the synthesised adsorbents.

2.6. Determination of the Adsorption of Chromium (VI) Ion from the Aqueous Solution

A 20 mL of chromium (VI) of 40 ppm solution was added to 0.5 g of RSU-1, RSU-2, and RSU-3. The mixture was covered and equilibrated on the orbital shaker at 300 for 60 minutes at a constant speed.

The mixture was filtered, and the residual chromium (VI) concentration was determined using the AAS. The procedure was carried out to determine the rate of percentage removal of the chromium ion by the individual adsorbents.

2.7. Determination of the Effect of Contact Time

A 20 mL, 40 ppm chromium (VI) solution was added to 0.2 g of RSU-1, RSU-2 & RSU-3. The mixture was covered and equilibrated on the orbital shaker at a constant speed of 300 rpm for 60 minutes.

The mixture was filtered, and the residual chromium (VI) concentration was determined using the AAS. The procedure was repeated at 90 mins, 120 mins, 150 mins and 180 mins to determine the effect of the contact time of the chromium ion with the individual adsorbents.

2.8. Determination of the Effect of Concentration of Chromium (VI) ion on Adsorption

20 mL of 40 ppm chromium (VI) solution was added to 0.2 g of the synthesised adsorbents. The pH of the mixture was 5.0. The mixture was covered and equilibrated on the orbital shaker at a constant speed of 300 rpm for 60 minutes. The mixture was filtered, and the residual chromium (VI) concentration was determined using the AAS. The procedure was repeated using 10, 20, 40, 60 and 100 ppm chromium (VI) ion concentrations for all the synthesised adsorbents.

2.9. Adsorption Isotherms

A concentration of 0.2 g of the adsorbents was used to obtain the adsorption isotherm data by varying the chromium (VI) ion concentration from 10, 20, 40, 60 to 100 ppm. Subsequent experimental procedures were observed, and the residual chromium (VI) concentration was determined using AAS. Isotherm data for RSU-1, RSU-2 & RSU-3 were generated. Langmuir and Freundlich isotherm models in equations 3 and 4 were used to interpret the adsorption process.

$$\frac{Ce}{qe} = \frac{Kl}{qmax} + \frac{1}{qmax} \tag{3}$$

$$\operatorname{Log} q_{e} = \log k_{f} + \frac{1}{n} \log C_{e}$$
(4)

Where V_{max} is the maximum adsorption at monolayer (mg/g). C_e is the equilibrium concentration of metal ions in solution.

 Q_e is the amount of ion adsorbed per unit weight of the adsorbent.

 K_L is the Langmuir constant related to the affinity of binding sites.

K_f and n are called Freundlich constants.

 $K_{\rm f}$ is the relative adsorption capacity, while n shows the adsorption intensity.

Adsorption isotherm is a feature that explains the extent of equilibrium in the relationship between the adsorbate in the solution and the adsorbate on the adsorbent [8].

The correlation between the amount of chromium (VI) ion adsorbed onto RSU-1, RSU-2 and RSU-3 was evaluated using two-parameter isotherm models. The experimental data acquired were analysed using Langmuir (equation 3) and Freundlich (equation 4) models.

The Langmuir isotherm model is employed based on monolayer adsorption of adsorbate on homogeneous active sites; hence, saturation is attained beyond which adsorbate is not attached to adsorbent [9].

The Freundlich model is another example of the twoparameter model applied based on multilayer adsorption on heterogeneous surfaces [10]. Consequently, Langmuir and Freundlich are all represented by specific constants whose values depict the surface properties of the adsorbent.

2.10. Adsorption Kinetics

The kinetic properties of adsorption were investigated by adding 0.2 g of the synthesised adsorbents to 20 mL of 40 ppm chromium (VI) solution and was agitated for 60, 90, 120, 150 and 180 minutes.

Other experimental procedures were followed equally, and the residual chromium (VI) concentration was obtained using AAS. The kinetic data for RSU-1, RSU-2 and RSU-3 were obtained.

The kinetics process was also analysed using Pseudo-firstorder (equation 5) and pseudo-second-order (equation 6) adsorption models.

$$Log (qe - qt) = log qe - (\frac{\kappa_1}{2.303}) t$$
 (5)

$$\frac{t}{qt} = \frac{1}{K2qe2} + \frac{1}{qe}t$$
(6)

In equations 5 and 6, qe and qt denote the amount of chromium (VI) ion adsorbed at equilibrium and at a time, t. K_1 and K_2 are the rate constants of the pseudo-first and pseudo-second order adsorption processes, respectively.

Pseudo-first and second-order models create a kinetic description of the adsorption process.

Conformity with the former and latter reflects physisorption and chemisorption mechanisms.

3. Results and Discussion

3.1. The Synthesised Adsorbents

The synthesised RSU-1, RSU-2, and RSU-3 were obtained as colourless crystals, as shown on plates 1, 2 and 3.



Plate 1. RSU-1 Crystals



Plate 2. RSU-2 Crystals



Plate 3. RSU-3 Crystals

3.2. Adsorbent Surface Morphology 3.2.1. Powder X-Ray Diffraction (PXRD).

The crystallographic structure of the RSU-1, RSU-2 & RSU-3 was studied. Powder x-ray diffraction micrographs of the synthesised adsorbent are presented in Figures 1, 2, and 3.



Fig. 1 Powder X-ray Diffraction (PXRD) Pattern of RSU-1 with Peaks



Fig. 2 Powder X-ray Diffraction (PXRD) Pattern of RSU-2 with Peaks



3.3. Adsorption study of chromium(VI) ion using RSU-1, RSU-2 and RSU-3

3.3.1. Effect of pH on Adsorption of Chromium (VI) ion

The result of pH on the adsorption of chromium (VI) ion onto the RSU-1, RSU-2, and RSU-3 is presented in Figure 4.





The results showed that the adsorption capacities of Cr (VI) ions on the adsorbents were positively dependent on the pH of the medium. As the pH (pH2 - pH10) tends to be alkaline, there was an increase in adsorption of Cr (VI) from 2,240 to19 580 mg/g for RSU-1, from 1,550 to 14,110 mg/g for RSU-2; and from 8,940 to 15,900 mg/g for RSU-3.

3.3.2. Rate of Percentage Removal of Chromium (VI) Ion by RSU-1, RSU-2 & RSU-3

The rate of percentage removal of chromium (VI) ion determined showed an increase in the rate of removal. Thus implying the adsorption of some amount of the chromium (VI) ion onto the adsorbent.



SYNTHESISED ADSORBENT Fig. 5 Rate of percentage Removal of chromium (VI) by adsorbent





Fig. 6 Plot of Effect of Contact Time on RSU-1, RSU-2 & RSU-3

The effect of varying contact time is shown in Figure 6. The time-dependent behaviour of Cr (VI) ion adsorption was tested by adjusting the contact time from 60-180 mins. The result showed that the adsorption capacity positively depends on the contact time. An increase in contact time from 60-180 mins increased the adsorption rate from 3340 to 7242 mg/g for RSU-1, 6155 to 9972 mg/g for RSU-2 and 3750 to 4880 mg/g for RSU-3. The adsorption capacity remained constant at 180 mins.

3.3.4. Effect of Concentration on Adsorption of Chromium (VI) Ion

The plots of adsorption capacity against chromium (VI) ion for RSU-1, RSU-2, and RSU-3 are revealed in Figure 7. There was an increase in adsorption capacities from 923.7 to 10,121 mg/g, 1650 to 10,570 mg/g, and 967.0 to 11,873 mg/g for RSU-1, RSU-2, and RSU-3, respectively as the concentration increased from 10 to 100 mg/l.



Fig. 7 Plot of Effect of Concentration on RSU-1, RSU-2 & RSU-3

 Table 1. Langmuir and Freundlich Isotherm Parameters of Chromium

Langmuir			Freundlich	
Adsorbent K _L (l\mg)	Qmax R ²		K _F (mg∖g) R ²	n
RSU-1 0.8431	-3,333.3	-41	21.1641 0.9375	0.5910
RSU-2 0.6585	-20,000	-118	34.0 X 10 ⁵ 0.8637	-0.3163
RSU-3 142 0.14	20,000 01	-	69.6787 0.8595	0.7464

3.4. Characterisation of Synthesised Adsorbent Powder X-Ray Diffraction (PXRD)

The result of the X-ray diffraction of RSU-1, RSU-2, and RSU-3 shows high-intensity Bragg diffraction peaks. The presence of these peaks confirms the crystalline property of the synthesised adsorbents, a notable characteristic expected of any metal-organic framework adsorbent.

3.5. Effect of pH on The Adsorption Capacity of the Adsorbents

The effect of pH on the adsorption capacity of the synthesised adsorbent was determined. There was an appreciable increase in the adsorption rate as the pH increased (from an acidic medium to an alkaline medium). The result of pH on the adsorption of chromium (VI) ions onto the adsorbents (RSU-1, RSU-2, and RSU-3) is presented in Figure 4. The results showed that the adsorption capacities of Cr (VI) ions on the adsorbents were positively dependent on the pH of the medium. As the pH tended to be alkaline (pH2 - pH10), there was an increase in adsorption of Cr (VI) from 2,240 to19 580 mg/g for RSU-1, from 1,550 to 14,110 mg/g for RSU-2; and from 8,940 to 15,900 mg/g for RSU-3. The better performance of the MOFs at basic pH is based on a decrease in the competition between the proton (H⁺) and the negatively charged surface of the adsorbent [11].

Adsorption increases with increases in pH, which may be because, at higher pH values, the surface of the adsorbent becomes negative, which enhances the adsorption of positively charged cations through the electrostatic force of attraction [12].

This may be attributed to the effect of H^+ ions in an acidic medium. These ions surround the surface of the adsorbent molecule, thereby limiting the adsorption of these surfaces. On the other hand, the observed increase in adsorption in the basic medium may be due to the decrease in H^+ ions, thereby making the adsorption sites more accessible to the adsorbents. The reason for the increase may also be because, in an acidic medium, heavy metals become mobile and adsorption onto the adsorbent becomes less effective due to saturation of the sorption site by the H^+ ion. Generally, sorption increases with pH [13, 14].

3.6. Rate of Percentage Removal of Chromium (VI) ion by the Adsorbents

The rate of percentage removal by the synthesised adsorbents (RSU-1, RSU-2, and RSU-3) is presented in Figure 5. The rate of percentage removal of Cr (VI) ion was highest with RSU-1, with a record of 41.66%. This was followed by RSU-3, which had a 27.08% removal rate, while the RSU-2 had a 14.58% removal rate of the Cr (VI) ion.

The difference in the percentage rate of removal of Cr (VI) ion may be attributed to the ionic size charge of the adsorbents. This is because ionic size charge determines the extent to which an element is solvated. Therefore, a small ionic charge had the highest percentage of removal among the three adsorbents studied.

3.7. Effect of Contact Time

The effect of varying contact time is shown in Figure 6. The time-dependent behaviour of Cr (VI) ion adsorption was tested by adjusting the contact time from 60-180 mins. The result showed that the adsorption capacity of Cr (VI) increased as the contact time increased.

This is attributed to the availability of the sorption site for more heavy metal adsorption with a rise in the contact time, indicating that the adsorption process is highly timedependent. However, as the contact time increased to 180 minutes, the adsorption reached the equilibrium point, and this is a situation where there is no vacant site available for more adsorption to occur, hence making the adsorption process constant as the sites are less available and the capacity of the adsorbent to adsorb more chromium (VI) ion get exhausted [15, 16].

This can also be due to the aggregation of the metal ion on the adsorbent with increased contact time, making it almost impossible to diffuse deeper onto the adsorbent structure at higher energy sites [17]. The aggregation negates the influence of contact time as the pore gets filled up and starts exhibiting resistance to the aggregated metallic ion's diffusion onto the adsorbent.

3.8. Effect of Concentration on Adsorption of Chromium (VI) Ion

There was an increase in adsorption capacities as the concentration of the adsorbate increased from 923.7 to 10,121 mg/g, 1650 to 10,570 mg/g, 967.0 to 11,873 mg/g for RSU-1, RSU-2 & RSU-3 respectively as the concentration increased from 10 to 100 mg/l.

This observation could be attributed to the fact that the saturation point is not yet attained and could still bind more chromium (VI) and can also imply that at higher concentration, the active site would still be surrounded by more of the chromium (VI), hence enhancing the adsorption process.

3.9. Isotherm Properties of the Adsorbents

The evaluated adsorption isotherm parameters in Table 1 are based on Langmuir and Freundlich plots. Correlation coefficient (R^2) values of 0.9375, 0.9831 and 0.8595 recorded for RSU-1, RSU-2, and RSU-3, respectively, indicate good linearity with Freundlich showing that the present data are in agreement with the Freundlich equation.

The RSU-3 show low correlation values (0.1401), indicating non-conformity with Langmuir, while RSU-1, with a correlation coefficient of 0.8431, show good linearity. The result, therefore, suggests multilayer adsorption for RSU-1 on heterogeneous adsorbent surfaces. Other parameters, such as Freundlich constant n, also provided information regarding the favourability of adsorption. The values of n for RSU-1, RSU-2 and RSU-3 are 0.5910, 0.3163 and 0.7464

respectively. This satisfies the condition of 0 < n < 1 for favourable adsorption.

4. Conclusion

The result indicates that the metal salts sodium, zinc and magnesium can be effectively employed as metal-organic framework adsorbents at their distinct capacities.

The adsorbents exhibited outstanding peaks when analysed using the powder X-ray diffraction technique. The occurrence of the peaks displays their crystalline properties, which is an expected characteristic of any credible MOF. BET analysis was also carried out, and it was observed to possess an appreciable large surface area, another characteristic expected of any credible MOF adsorbent.

The adsorption rate of the synthesised adsorbents' Cr (VI) ions increased with contact time, pH and concentration.

The ionic size charge of adsorbents affects the rate of percentage removal of the adsorbates in the order of RSU-1> RSU-3> RSU-2.

The adsorbents synthesised are insoluble in organic solvent (N-hexane) but soluble in an acidic medium (Sulphuric acid).

Freundlich model gave the best fit with the adsorption data generated, thus suggesting a heterogeneous coverage of good adsorption from the solution. The kinetic data fit suitably with the Pseudo-second order model, implying chemisorption.

The investigation supports and encourages the usage of adsorbent with the metal-organic framework.

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