# Hydrothermal Synthesis, Characterization and Adsorption Testing of $MoS_2$-Zeolite for the Removal of Lead in an Aqueous Solution

Elvis Fosso-Kankeu*, Martin Joseph Spiro, Frans Waanders, Neeraj Kumar, Suprakas Sinha Ray and Nico Lemmer

***Abstract*—The shortage of water can be worsened by the pollution of limited water resources by industrial activities such as mining which contribute to significant level of toxic heavy metals in the environments. Heavy metals such as lead could negatively affect the health of consumers ingesting contaminated water and must therefore be removed from existing water sources to ensure that these sources can be used effectively and safely.**

**In this study the potential of zeolite (clinoptilolite) and molybdenum sulfide as effective adsorbents and lead-selective adsorbent, respectively was considered for the hydrothermal synthesis of $MoS_2$-Zeolite composite for effective removal of lead from aqueous solution. The synthesized composite and the parent compounds were characterized using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and Fourier transformed infrared spectroscopy (FTIR). The results confirmed the properties of the adsorbents as well the successful synthesis of the composite. The adsorbents were used for the removal lead from solution while assessing the effect of adsorbent dosage and initial concentration of lead on the adsorption performance. It was found that clinoptilolite, $MoS_2$ and $MoS_2$-zeolite exhibited adsorption capacities of 3.45, 4.1 and 1.2 mg/g, respectively; indicating that $MoS_2$ was the superior adsorbent. This implies that for metal contaminated solutions, $MoS_2$ will be the ideal adsorbent for the removal of lead.**

***Keywords*—** Water pollution, lead, adsorbents, zeolite, molybdenum sulfide, adsorption kinetics

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E. Fosso-Kankeu is with the Water Pollution Monitoring and Remediation Initiatives Research Group in the School of Chemical and Minerals Engineering of the North West University, South Africa.

F. Waanders is with the Water Pollution Monitoring and Remediation Initiatives Research Group in the School of Chemical and Minerals Engineering of the North West University, South Africa.

Martin J. Spiro is with the Water Pollution Monitoring and Remediation Initiatives Research Group in the School of Chemical and Minerals Engineering of the North West University, South Africa.

Nico Lemmer is with the Water Pollution Monitoring and Remediation Initiatives Research Group in the School of Chemical and Minerals Engineering of the North West University, South Africa.

Neeraj Kumar is with the DST-CSIR National Centre for Nanostructured Materials, Council for Scientific and Industrial Research, Pretoria 0001, South Africa.

Suprakas S. Ray is with the DST-CSIR National Centre for Nanostructured Materials, Council for Scientific and Industrial Research, Pretoria 0001, South Africa.

## I. Introduction

The recent water crisis in Cape Town, South Africa, contributed to the decrease of crops yields, wine shortage, water restrictions for households and industries and a decline in tourism which affected to a certain extent the economy of the country [1-3]. Reduced rainfall, over-exploitation of water sources, failing infrastructure and water pollution also contribute to water shortages, hence the need to consider return flow through decontamination of polluted water.

Heavy metals such as lead, cadmium, mercury and arsenic are the primary heavy metals that pose a threat to human [4]. International bodies such as the World Health Organization review the dangers of exposure to these metals regularly and many studies are done that aim to reduce exposure to heavy metals [5] and irrespective of the adverse effects that these metals have on humans health, exposure to these heavy metals have been recurrent and even increased in less developed countries [6]. More developed countries have however been reducing the contamination of water sources over the past 100 years thus reducing the exposure to heavy metals [5, 6].

There are different treatment processes for water contaminated with heavy metals such as precipitation, membrane filtration, ion exchange, adsorption, co-precipitation/adsorption and many others. Most of these techniques are expensive, energy intensive, produce harmful by-products or are difficult to implement safely [7-15].

Chemical precipitation, for example, is inefficient with regards to time consumption, poor settling, aggregation of metal precipitates and the long-term impact of sludge disposal as a lot of sludge by-product are produced [16]. Ion exchange using synthetic organic ion exchange resins cannot handle concentrated metal solutions as the ion exchange resin matrix gets fouled easily. Ion exchange is also non-selective and is highly sensitive to pH [7].

Adsorption is the most popular technique used for the removal of heavy metals from waste streams [17-28]. Adsorption involves mass transfer of particles from liquid phase to the surface of a solid where it is bound by physical and/or chemical interactions. The most frequently used adsorbent is activated carbon, which, despite its wide range of industrial applications, is still an expensive material [16].

Clinoptilolite is the most commonly found natural zeolite with the chemical formula $(K_2Na_2Ca)_3Al_6Si_{30}O_{72}{\cdot}21H_2O$ (Wang

& Peng, 2010). It is one of the most frequently studied natural zeolites due to its heavy metal selectivity series which was determined to be $Pb^{2+} > Cd^{2+} > Cu^{2+} > Co^{2+} > Cr^{3+} > Zn^{2+} > Ni^{2+} > Hg^{2+}$ with the ion exchange equilibrium being favourable for $Pb^{2+}$ [29].

Considering the affinity that molybdenum sulphide naturally has for lead [30] and combining it with a zeolite such as clinoptilolite which shows a varying adsorption capacity for lead depending on the region where it was found [31] may lead to a $MoS_2$-zeolite compound that shows a heightened affinity for lead as well as a greater adsorption efficiency.

This study focuses on $MoS_2$-zeolite compound acting as a cheap and effective adsorbent alternative to activated carbon.

## II. MATERIALS AND METHODS

### A. Materials and glassware

Lead-water samples were prepared using lead nitrate ($N_2O_6Pb$) with a total molecular mass of 331.21 g/mol. The percentage of lead in the lead nitrate was 62.5%.

Molybdenum (IV) sulfide was used along with clinoptilolite zeolite to form the $MoS_2$-zeolite complex for use as an adsorbent and were each also tested for their absorptivity.

Volumetric measurements were made using 2 syringes, one 20 mL and one 5 mL, as well as a 200 µL pipette and all solutions and mixtures were prepared in sterile Greiner Bio 50 mL CELLSTAR centrifuge tubes before being poured into 100 mL Simax® clear graduated borosilicate glass media bottles with blue polypropylene lids.

### B. Hydrothermal synthesis

One gram of $MoS_2$ was added to 20 mL of water and stirred for 30 min at $25^0$ C. Then 10 mL of a 50/50 glycerol-water solution was added to the mixture and mixed for another 10 min which resulted in the formation of $MoS_2$ vitreosol. At this point 0.5 g of zeolite was added to the suspension and stirred for another 30 min. All mixing up to this point were done at 500 RPM. Once the mixing was complete the mixture was placed in a high pressure and temperature resistant ceramic vial which was then placed inside a steel securing tube. The tube was screwed shut tightly and then placed inside an autoclave that was set at $140^0$ C and was left inside for 3 hours for the synthesis to take place (generally heating of the autoclave takes 30 min extra). The hot metal tube was left to cool for another 2 hours after which the synthesized solution was poured into a centrifuge tube and the centrifuge run at 8500 RPM for 5 min. The liquid was decanted off the solid and the centrifuge tube was filled to 50 mL with deionised water, shaken by hand and then centrifuged at 8500 RPM for 5 min again, this was done twice to ensure product purity. The wet solid was dried at $80^0$ C in the oven for a minimum of 5 hours until dry. The dried powder was then weighed, and the process was repeated until 5 g of powder was obtained with 1 g of powder being used for analysis.

A powder sample of each of the adsorbents was then sent for analysis using FTIR and SEM-EDS to aid in proper characterization of the particles.

### C. Lead solutions

The lead stock solution of 1000 ppm concentration was prepared using lead nitrate salt that was dissolved in deionized water. A corresponding amount of the stock solution was used to prepare working solutions of a given concentration.

### D. Adsorption tests

The first adsorption test was to determine the effect of adsorbent dosage on the adsorption of lead. This was done by applying 0.025 g, 0.050 g and 0.075 g of adsorbent to each 25 mL lead/water solution (60 ppm). Each dosage of the respective adsorbent clinoptilolite (zeolite), $MoS_2$ and $MoS_2$-zeolite complex, was tested in duplicate. The dosed solutions were incubated in a Labcon shaking incubator and mixed at 160 RPM and $25^0$ C for an hour. The samples were centrifuged at 4000 RPM for 10 minutes to separate the remaining lead/water solution from the adsorbent. The decanted lead/water solution was sent for analysis by Inductively coupled plasma-atomic emission spectrometry (ICP-AES) to determine the adsorption efficiency of the adsorbent.

Once the optimum adsorbent dosage was determined the effect of the initial concentration of lead was tested by preparing 6 samples each of 1, 1.5, 3.5, 6, 8.5, 10 and 12.5 ppm lead-water solution and adding the necessary dosage of adsorbent. After allowing adsorption to take place in an incubator for 1 hour at $25^0$ C the samples were centrifuged to separate the solids from the solution and the solution was tested for the concentration of lead remaining in each case. A control sample of each lead dosage was sent for analysis to ensure that the correct lead concentrations could be considered during the calculation of the amount adsorbed.

## III. RESULTS AND DISCUSSION

### A. Particle morphology (SEM-EDS analysis)

The following images depict the SEM-EDS analysis of the adsorbents used in the experimental procedures.

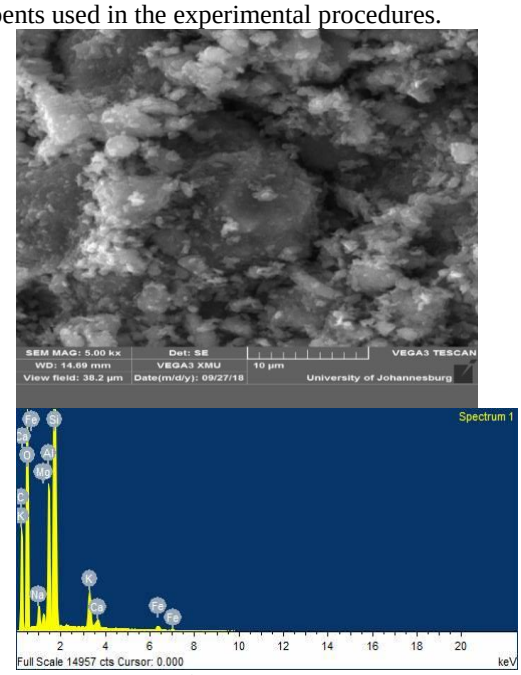


Fig. 1. SEM-EDS analysis of clinoptilolite zeolite showing its structure and composition.

Figure 1 depicts the SEM-EDS analysis of clinoptilolite, the zeolite used in the hydrothermal synthesis and as an adsorbent. The SEM images show a series of oval beads which provide a lot of surface area on which adsorption can take place while the EDS confirms that the zeolite is an aluminosilicate as seen from the higher amount of silicon (Si) and aluminum (Al).

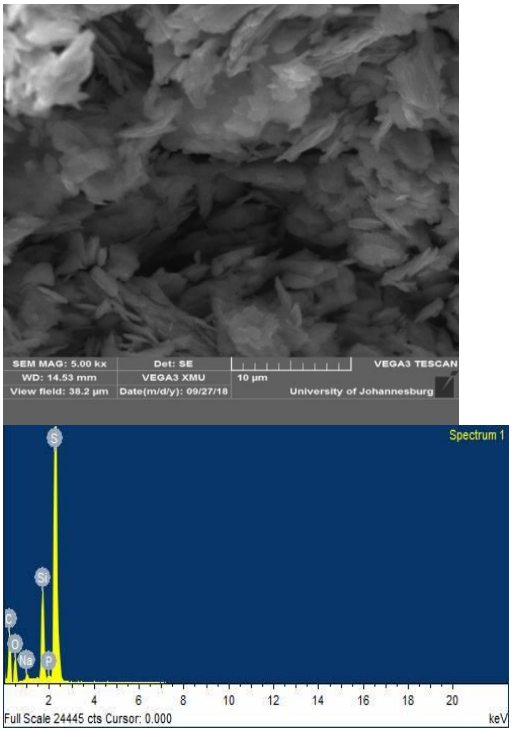


Fig. 2. SEM-EDS analysis of MoS2 showing its structure and composition.

Figure 2 depicts the SEM-EDS analysis of $MoS_2$, the second component for the hydrothermal synthesis and one of the adsorbents used in the experiments. The SEM images show a series of pine-like structures which provide maximum surface area on which adsorption can take place while the EDS shows the dominance of the primary component of $MoS_2$ which is sulfur (S).

Figure 3 depicts the SEM-EDS analysis of $MoS_2$-zeolite, the hydrothermally synthesized adsorbent. The SEM structures appear as clouds that combine both the pine-like structures of $MoS_2$ and the beads structures from clinoptilolite. The EDS shows that the elements (aluminum, silicon and Sulphur) found in clinoptilolite and $MoS_2$ are all present in the $MoS_2$-zeolite structure implying that the hydrothermal synthesis of the adsorbent was successful.

### *B. Polymer binding groups (FTIR analysis)*

The following FTIR graphs represent the analysis of clinoptilolite, $MoS_2$ and $MoS_2$-zeolite.

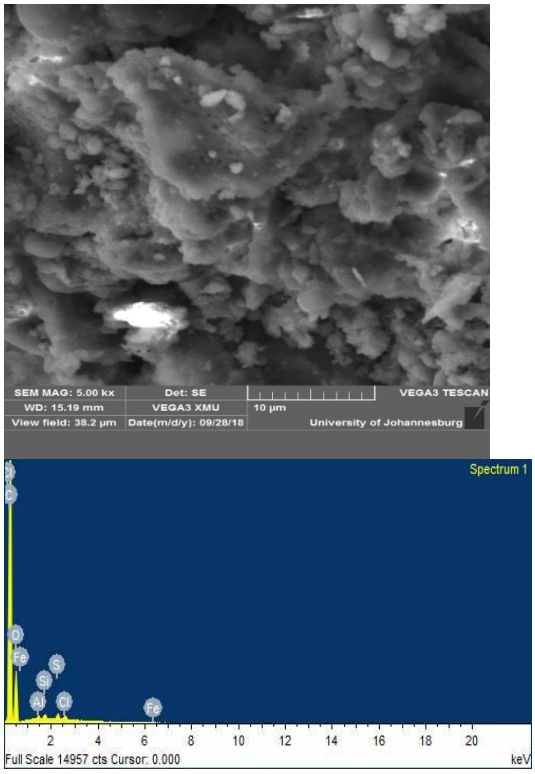


Fig. 3. SEM-EDS analysis of MoS2-zeolite showing its structure and composition.

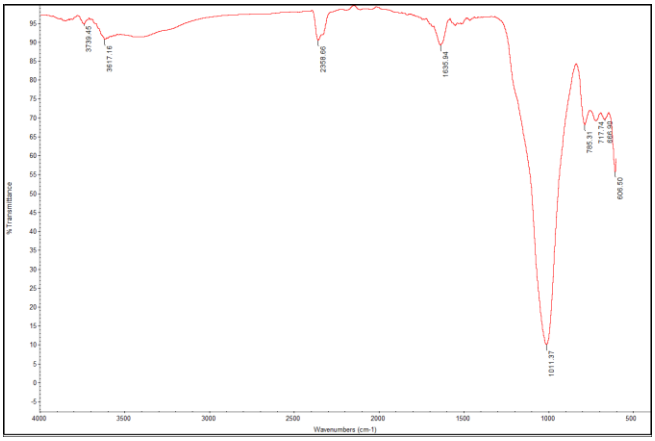


Fig. 4. FTIR analysis of clinoptilolite showing infrared peaks.

The spectrum of clinoptilolite in Figure 4 shows a peak between 3700 and 3584 $cm^{-1}$ and a peak between 1635 and 1648 $cm^{-1}$ which would represent medium strength O-H bonds due to the presence of water in the natural zeolite. A very strong peak between 1000 and 1400 $cm^{-1}$ which is closer to 1030 $cm^{-1}$ represents bending vibrations of Si-O or Al-O bonds in the clinoptilolite structure [32].

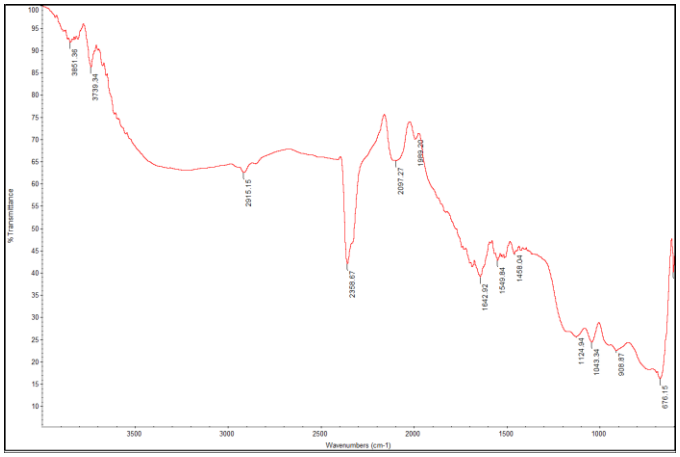

Fig. 5. FTIR analysis of MoS2 showing infrared peaks.

$MoS_2$ spectrum in Figure 5 shows two peaks at very high wavelengths that are representative of medium O-H, due to water present in the structure of $MoS_2$; adsorbance peaks of S=O and Mo-O were observed at wavelengths 1124.94 and 676.19 cm-1, respectively [33].

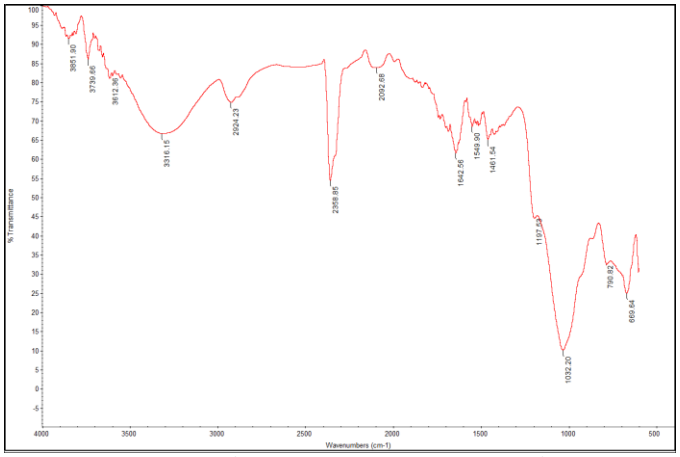

Fig. 6. FTIR analysis of MoS2-zeolite complex showing infrared peaks.

The $MoS_2$-zeolite spectrum in Figure 6 has more infrared peaks than the clinoptilolite and the $MoS_2$, which obviously shows the combination of both; more important is the peak at 1032.20 $cm^{-1}$ which represents bending vibrations of Si-O or Al-O bonds in the clinoptilolite structure, while the peaks at wavelengths 1197.53 and 669.64 cm-1 represent S=O and Mo-O groups respectively.

### C. *Effect of adsorbent dosage on lead removal*

Three dosages of each of the three adsorbents were added to 60 mg/L lead-water solution and after centrifugation the remaining liquid was tested for its lead content. The following graph shows the effect of the different adsorbent dosages, being 0.025 g, 0.05 g and 0.075 g, on the % of lead removed from the water.

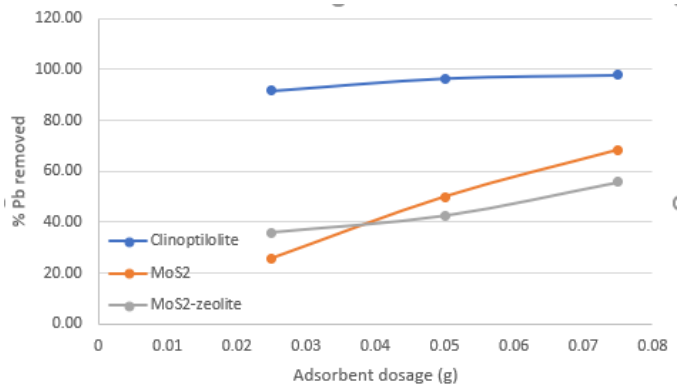


Fig. 7. The % of lead removed by the adsorbents based on adsorbent dosage.

From Figure 7 it can be seen that the % of lead removed by the clinoptilolite was close to 100% regardless of the dosage used.

The percentage of lead removal by the other two adsorbents appeared to be low but also increased as the dosage increased. It was thus concluded that the optimal dosage for future experiments should be the highest dosage of 0.075 g.

### D. *Effect of lead concentration in the solution*

The effect of lead dosage in the water was tested by making lead-water solutions of different lead concentrations and adding 0.075 g of each adsorbent to test how much of the lead each adsorbent was able to remove.

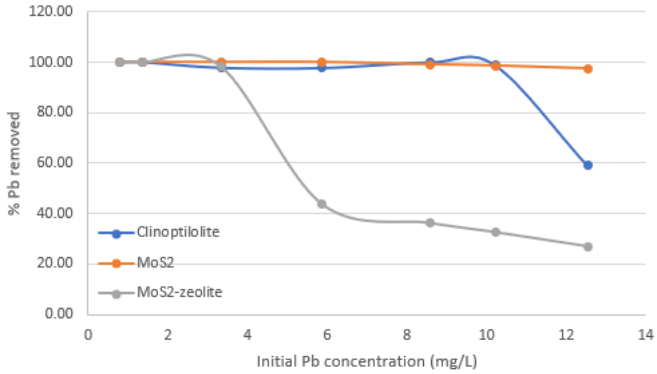


Fig. 8. Graph showing the effect of lead dosing on the % of lead removed from the solution.

In Figure 8 it can clearly be seen that each of the adsorbents removed close to 100% of the lead from the water, however the $MoS_2$-zeolite became saturated very quickly while clinoptilolite was saturated at much higher lead dosages. On the other hand, the adsorption performance of $MoS_2$ was constant throughout the ranges of lead concentrations, implying higher adsorption capacity.

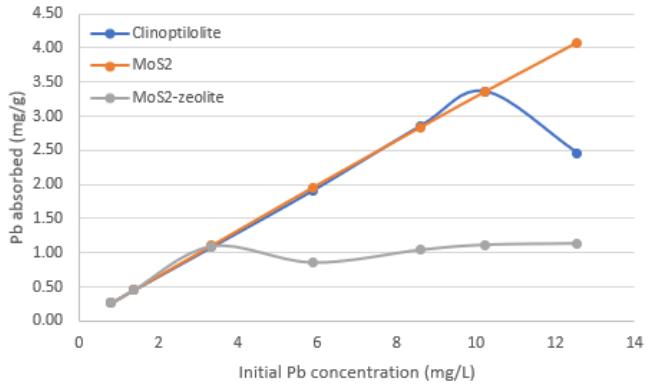


Fig. 9. The effect of initial lead concentration on the amount of lead adsorbed in mg per gram of adsorbent.

Figure 9 is a depiction of the effect of the initial lead concentration on the amount of lead adsorbed onto each adsorbent. However, this figure clearly shows the adsorption capacity of each adsorbent; it can be therefore observed that the adsorption capacities of clinoptilolite, $MoS_2$ and $MoS_2$-zeolite are 3.45, 4.1 and 1.2 mg/g, respectively. This implies that clinoptilolite and $MoS_2$ exhibit better adsorption capacities compared to $MoS_2$-zeolite; there is a slight advantage for $MoS_2$ which has been reported to have high affinity for Pb. Although it was expected that the composite $MoS_2$-zeolite will perform better, it could be suggested that the hydrothermal synthesis resulted in the reduction of the porous structure and ion exchange capacity of clinoptilolite due to the binding of $MoS_2$ at its surface, while the surface of $MoS_2$ trapped in the pores of clinoptilolite did not also contribute to the adsorption, leading to the hindrance of the potential benefit of combination of the two adsorbents.

## IV. Conclusion

Hydrothermal synthesis was successfully used to synthesize a $MoS_2$-zeolite complex which exhibited combined physico-chemical properties from the parent compounds. The use of the newly synthesized $MoS_2$-zeolite complex as well as $MoS_2$ and clinoptilolite for the removal of lead from solution showed that the complex had just lower adsorption capacity compared to the parent compounds, implying that $MoS_2$ and clinoptilolite should be considered individually for effective removal of lead from solution.

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**M. J. Spiro** was born in Johannesburg, South Africa in 1993 and was a Chemical Engineering student at the North West University (NWU) Potchefstroom campus.

He spent much of his time participating in engineering projects such as the completion of a literature study for a biodiesel plant through the THRIP student program as well as assisting in the construction of the SUNfarming Food & Energy Training Centre in South Africa that is located on the NWU engineering campus.