

# Removal of Ni<sup>2+</sup> and Pb<sup>2+</sup> IONS from Aqueous Medium Using Unmodified and 4-Aminophenol Modified Red Onion Skin Extract

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## Abstract

The extraction of Ni<sup>2+</sup> and Pb<sup>2+</sup> ions from aqueous medium using unmodified (ROSE) and the 4-aminophenol modified red onion skin extract (ROSEDS) has been successfully achieved. Cold solvent extraction technique using a 50:50 acetone/water ratio was used for extraction of the red onion skin at 125 µm mesh size. The metal concentrations were determined using Atomic Absorption Spectrometry technique. Optimum percentage removal of the metal ions was achieved at pH 6.26 for Ni<sup>2+</sup> (89.40 % for ROSE and 97.60 % for ROSEDS) and pH 7.57 for Pb<sup>2+</sup> (80.10 % for ROSE and 82.56 % for ROSEDS). Optimum adsorption capacities of ROSE and ROSEDS for Ni<sup>2+</sup> were 6.30 mg/g and 7.10 mg/g respectively; while for Pb<sup>2+</sup> ions, values obtained for ROSE and ROSEDS were 5.87 mg/g and 6.34 mg/g respectively. Varying adsorbents dosage at fixed metals ion concentration yielded optimum percentage removal of 89.90 % and 97.90 % for Ni<sup>2+</sup>, while 86.85 % and 90.80 % for Pb<sup>2+</sup>, using ROSE and ROSEDS, respectively. Adsorption capacities of the adsorbents varied inversely with adsorbent dosages. In general, 4-aminophenol modified red onion skin extract (ROSEDS) gave comparatively, better results for the extraction of Ni<sup>2+</sup> and Pb<sup>2+</sup> ions from aqueous medium; relative to the unmodified red onion skin extract (ROSE). Additionally, extraction of Ni<sup>2+</sup> ions gave higher percentage removal values in both adsorbents when compared to extraction of Pb<sup>2+</sup> ion under similar conditions. This result has been attributed to differences in their ionic radii.

**Keywords:** ROSE, ROSEDS, Percentage Removal, Adsorption Capacity, Nickel (II) ion, Lead (II) ion.

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## INTRODUCTION

The desire for a more comfortable life for humans has given rise to natural environment exploitation leading to reduction in its capacities for self-stabilization (Horsfall & Spiff, 2013). Anthropogenic activities such as urbanization, agricultural and industrial development not only deteriorate the natural resources but also generate hazardous substances which lead to environmental pollution, global warming and acidic rain. Environmental problems have troubled mankind since the first man roamed the earth (Horsfall & Spiff, 2013). It is known to occur mostly in first world countries and the problem is gradually increasing by the day since the inception of industrial revolution. Large quantities of industrial effluents in developing countries are discharged with little or no treatment into the water bodies polluting the consumable water supply (Suleman, *et al.*, 2007). Industrial effluents, agricultural wastes and domestic sewages are the major substance

polluting the environment. Environmental pollution has so much become a popular topic worldwide, such that no new day passes without some discussion being centred on the issue.

The scarcity of safe and clean drinking water is one of the most pressing issues menacing the world's nations, particularly those in the developing world (Ramakrishna *et al.*, 2015). This challenge is an attendant effect of the industrialization of chemical industries, which results in the release of effluents containing a huge amount of organic and inorganic pollutants (laden with heavy metals) into the environment (Renu *et al.*, 2017, Ramakrishna *et al.*, 2015). They could be either natural (products of some natural processes) or man-made (as a result of human activities); biodegradable (decomposed by microorganisms) or non-biodegradable (not decomposed by microorganisms). Generally, water and environmental pollution by heavy metals is of great concern because the pollutants are biologically

indestructible; and persist in the aquatic and terrestrial environments. These pollutants accumulate over time to different level of toxicity in the flora and fauna systems. Therefore, sustenance of healthy life in our environment invariably requires the adequate treatment of the effluents to the recommended standard before discharging into the waterways.

Application of adsorption processes in the removal of poisonous metals from aqueous medium has been reported severally in different spheres of science and technology (Waweru *et al.*, 2016; Orji *et al.*, 2016; Chukwu & Manizabayo 2019). Using natural extracts of plants origin for removal of heavy metallic ions from aqueous medium is related to their affordability, reusability, stability, readily availability, eco-friendly, applicability and biodegradability. The adsorbent properties of the unmodified agricultural residue are due to the presence of carboxylic, phenolic and multiple hydroxyl groups in the substrates (Mcsweeny *et al.*, 2006; Liang, *et al.*, 2009; Hossain *et al.*, 2012). However, these adsorbents can undergo further modifications to increase their active binding-site(s) and also make them readily available for adsorption (Ibezim-Ezeani, & Okon, 2016; Ogali, *et al.*, 2008; Chukwu & Manizabayo 2019).

Onion (*Allium cepa*) is one of the world's oldest vegetables, grown widely and known for their characteristics, medical properties and their pungent flavours. Onion has three major types: yellow, white and red. It differs in colour as well as in size, flavour and shape. They are known to contain large amounts of dietary flavonoids especially quercetin (Schwartz and Mohan 2008; Slimestad *et al.*, 2007), this quercetin are found in much higher concentrations in the onion skin when compared to other parts such as fleshy bulb (Akoh & Sellappan, 2002; Shock *et al.*, 2012; Kim *et al.*, 2006). As an agricultural waste, red onion skin extract has been extensively used for adsorption and extraction studies (Chukwu and Manizabayo 2019; Chukwu & Maduabuchi 2023; Orji *et al.*, 2016; Akaranta & Effanga 1997). Researchers (Orji *et al.* 2016) have utilized extracts from red onion skin to remediate cadmium (II) ion ( $Cd^{2+}$ ) and Lead (II) ion ( $Pb^{2+}$ ) from their various aqueous solutions while Ibezim-Ezeani & Okon (2016) analysed the uptake of metal-ions anchored in aqueous medium using sorption mechanism by exploiting chemically modified onions skin extracts (*Allium Cepa* extract). In developing countries like Nigeria, large amounts of waste from red onion skin are generated daily which bring about environmental and disposal problems.

In continuation of our study on the extraction of  $Cu^{2+}$ ,  $Ni^{2+}$  and  $Zn^{2+}$  using 2-Aminophenol modified quercetin and red onion skin extract from Aqueous Medium (Chukwu & Maduabuchi 2023), this current research aims to at the adsorption studies in removing the metal ions  $Ni^{2+}$  and  $Pb^{2+}$  from aqueous medium

using unmodified and 4-aminophenol modified red onion skin extract.

## MATERIALS AND METHODS

### Collection and Preparation of Red Onion Skin

The red onion skin (ROS) were obtained from Omoku main market in Ogba/Egbema/Ndoni Local Government Area of Rivers State, Nigeria. The sample was carefully sorted and washed with deionized water and further air-dried for 10 days. The dried sample was pulverized using an electric milling machine and store at room temperature.

### Extraction procedure of Red Onion Skin (ROS)

Cold solvent extraction technique using a 50:50 acetone/water ratio was used for the extraction of 250 grams of the red onion skin (125  $\mu$ m). The extraction mixture was mechanically agitated for not less than six hours and thereafter allowed to stand for 24 hours before separation. The filtrate was concentrated to obtain a brown pasty extract called red onion skin extract (ROSE). The pasty extract (ROSE) was further allowed to dry in a desiccator for several days in order to obtain a dry form of ROSE.

### Modification of ROSE

The process of modification of ROSE was adopted from Chukwu & Maduabuchi 2023 with slight modification. In a 250 ml beaker, sodium hydroxide (NaOH) solution (10%) was prepared by dissolving 6.0 g of sodium hydroxide pellets in 54ml of distilled water. This was followed by the dissolution of 7.2 g of the pulverized ROSE in the 10% sodium hydroxide (alkaline) solution; and the resultant solution was kept in an ice bath.

### Preparation of Benzenediazonium Salt Solution (Diazotization)

Further, a known mass of dry sodium nitrite (1.3798 g) was dissolved in 10ml of cold deionized water and the solution was kept in an ice-bath for cooling. Thereafter, 2.401 g of 4-aminophenol were dissolved in 90 ml of cold distilled water in a 250 ml beaker. This was followed by the gradual addition of 24 ml of concentrated hydrochloric acid while stirring simultaneously, until the 4-aminophenol was completely dissolved, while the stirring continued in a cold ice bath. The cold sodium nitrite solution was then added in drops to the acidified 4-aminophenol, while continuously stirring the mixture for 3-5 minutes.

### Coupling of ROSE Solution and Benzenediazonium Salt Solution

The benzenediazonium salt solution was coupled with the red onion skin extract (ROSE) by slowly adding the benzenediazonium salt solution to the alkaline solution of red onion skin extract for 5 minutes. This was followed by stirring of the mixture at 0 °C for 30 minutes in order to achieve a complete reaction between the two solutions. At the end of the coupling

process, suction filtration was employed for the filtration of the mixture; and the residue on the Buchner funnel was washed with a small amount of cold water and dried for about 35 seconds with the suction turned on. The resultant product obtained was labelled ROSEDS and placed on a watch glass and allowed to dry for two days. Thereafter, the percentage yield was determined.

#### Preparation of Standard or Stock Metal Solutions

The stock solution of nickel ions ( $\text{Ni}^{2+}$ ) of 1000ppm was prepared by dissolving 4.24 grams of  $\text{Ni}(\text{CH}_3\text{COO})_2$  in 1.0 litre of deionized distilled water, while the stock solution of lead ( $\text{Pb}^{2+}$ ) ions was prepared by dissolving 0.795 grams of  $\text{Pb}(\text{NO}_3)_2$  in 0.5 litre of deionized distilled water. These solutions were diluted to obtain solutions of various known concentrations of lead and nickel. Working standard solutions of 15 mg/l of  $\text{Ni}^{2+}$  and  $\text{Pb}^{2+}$  ions were obtained by dilution of the respective stock solutions. All the reagents used were of analytical grade.

#### Preparation of Extractant

The extractant solution was prepared by dissolving the required concentration each of ROSE and ROSEDS in 200 ml of ethyl acetate in separate beakers.

#### Extraction of the selected heavy metals

A measured amount of 0.2 ml of,  $\text{Ni}^{2+}$  and  $\text{Pb}^{2+}$  solutions were poured into different sample bottles, secondly, 1.8 ml of the buffer solution of different pH were added to the sample bottles and lastly, 2 ml of the ROSE (extractant) was added to the mixtures in the sample bottles with the aid of a micro-pipette. A similar procedure was repeated for ROSEDS. The mixture was agitated for 45 minutes with the aid of a mechanical shaker, allowed to stand and separate into two phases. 1ml of the aqueous phase was taken from each mixture and placed in different sample bottles after which it was diluted with 2 ml distilled water to reduce its concentration. Each sample was analyzed using Perkin Elmer (AAnalyst 200) atomic absorption spectrophotometer (AAS). The amount of the metal ion extracted at its optimal pH was obtained by the difference between the initial metal ion concentration and the supernatant solution.

#### Effect of Variation of Dosage

The effect of dosage on the extraction of heavy metals was studied by agitating the mixture of 0.2 ml of each metal solution and 1.8 ml of the optimal value of pH of buffer and the solution of the extractant at different weights. Afterward 1ml of the aqueous

solution was diluted using 2 ml distilled water and sent for further analysis using the AAS.

#### Effect of Initial Metal Concentration

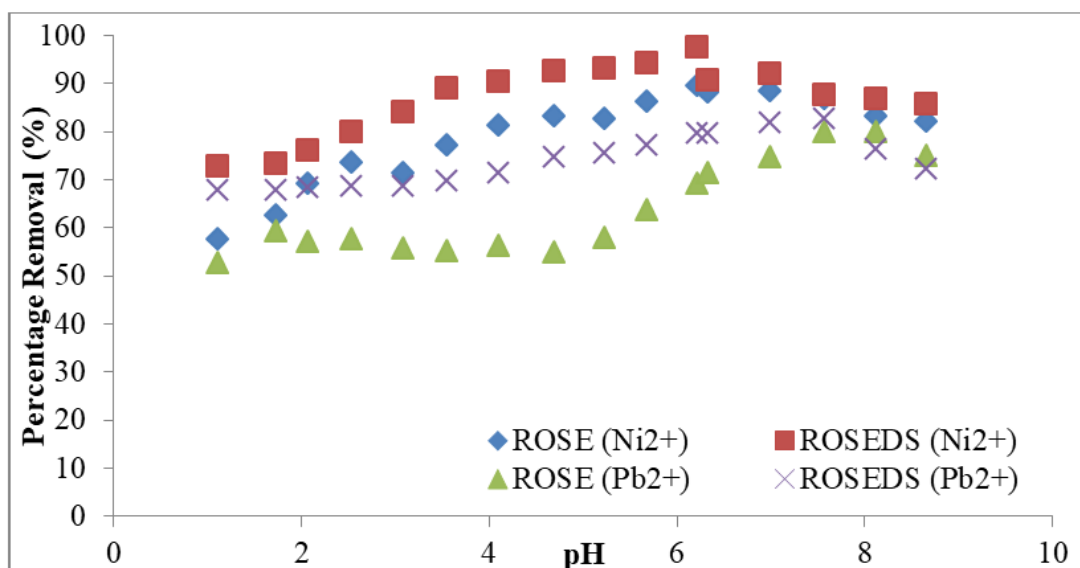
The effect of metal ion concentration on the extraction of heavy metals was studied by agitating the mixtures of 0.2 ml of metal ion concentration, 1.8 ml of each optimal pH of each of the metal and 2 ml of extract for 45 minutes, after which 1ml of aqueous solution was taken out using pipette, diluted to various concentration and afterwards sent for AAS analysis.

## RESULTS AND DISCUSSIONS

Cold solvent extraction technique using a 50:50 acetone/water ratio yielded the best result for extraction of the red onion skin at 125  $\mu\text{m}$  mesh size. Detailed solubility results of the ROSE in different solvents have been presented as published in Chukwu & Maduabuchi 2023. Similarly, results for melting points and other physicochemical properties of the ROSE have been reported and published (Chukwu & Maduabuchi 2023).

Furthermore, Figure 1 presents the results for determination of optimum pH for the removal of  $\text{Ni}^{2+}$  and  $\text{Pb}^{2+}$  ions from aqueous medium using unmodified (ROSE) and 4-aminophenol modified (ROSEDS) red onion skin extract as percentage removal plots of both metal ions.

From the results, 15.0 mg/l of  $\text{Ni}^{2+}$  ions at different pH values (1.0 to 9.0) using fixed mass of ROSE and ROSEDS showed a gradual increase in percentage removal from 57.4%, and 72.6% respectively to a maximum of 89.4% (ROSE) and 97.6 (ROSEDS) at a pH value of 6.21. Thereafter, further increase in pH resulted in a corresponding decrease in the percentage removal. Thus optimum pH for the percentage removal for  $\text{Ni}^{2+}$  using ROSE and ROSEDS is 6.21. Similarly, 15.0 mg/l of  $\text{Pb}^{2+}$  ions at different pH values (1.0 to 9.0) using fixed mass of ROSE and ROSEDS showed an increase in percentage removal from 52.7%, and 67.8% respectively to a maximum of 80.1% (ROSE) and 82.56 (ROSEDS) at a pH value of 7.57. Hence, optimum pH for the percentage removal for  $\text{Pb}^{2+}$  using ROSE and ROSEDS is 7.57. Generally, percentage removal of both metal ions ( $\text{Ni}^{2+}$  and  $\text{Pb}^{2+}$ ) was better using 4-aminophenol modified red onion skin extract (ROSEDS) in comparison to the unmodified ROSE. A similar observation had been reported for extraction studies using 2-aminophenol modified red onion skin (Chukwu & Maduabuchi 2023).



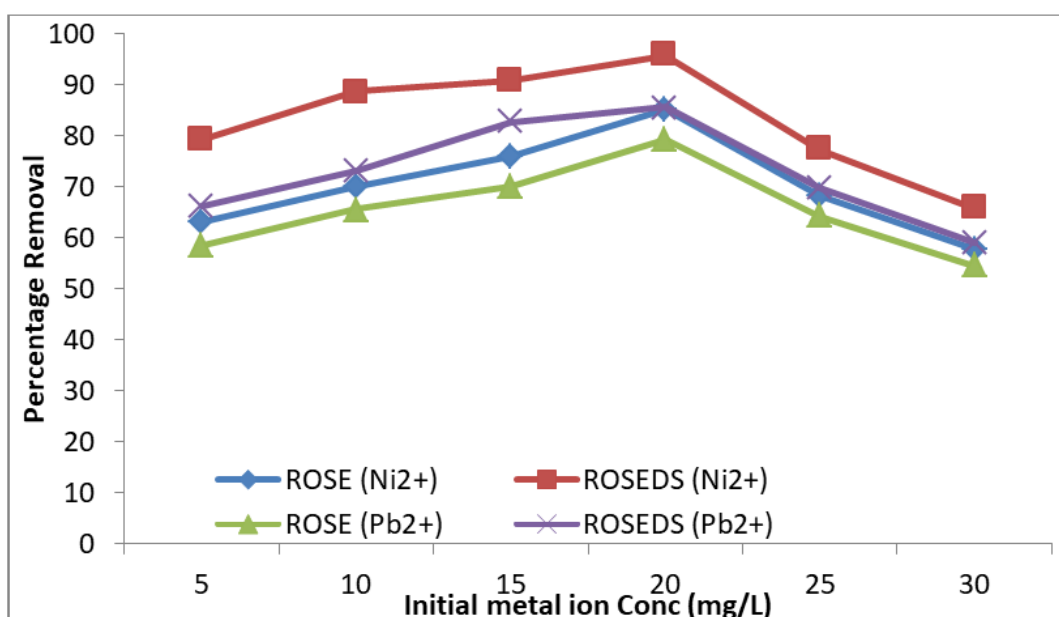
**Figure 1: Percentage Removal of Ni<sup>2+</sup> and Pb<sup>2+</sup> Ions at Different pH Values using fixed mass of ROSE and ROSEDS**

Figure 2 presents the results for variation in metal ion concentrations (mg/l) on the percentage removal (%) of Ni<sup>2+</sup> and Pb<sup>2+</sup> ions using a fixed mass (0.54g) of ROSE and ROSEDS at their optimum pH values.

The results of the study show that variation in metal ions concentration has positive effect on the percentage removal of the metal ions from aqueous medium of ROSE and ROSEDS. As the Ni<sup>2+</sup> ion concentration increased from 5.0 to 20 mg/l, the percentage removal of the metal ion by ROSE increased from 63.2% to 85.1%, while that of ROSEDS increased from 79.3% to 95.7% within the same range of metal

ion concentration. At 20.0 mg/l of the metal ion, the optimum percentage removal of Ni<sup>2+</sup> by ROSE and ROSEDS are 85.1% and 95.7%, respectively.

The same trend was observed for Pb<sup>2+</sup> ion using the same adsorbents. For ROSE, the percentage adsorption of Pb<sup>2+</sup> increased from 58.6% to 79.3%; while using ROSEDS, an increase from 66.2% to 85.6% was observed as the metal ion concentration increased from 5.0 to 20.0 mg/l. The optimum percentage removal of the metal ion for ROSE and ROSEDS were 79.3% and 85.6%, respectively, at 20.0 mg/l.



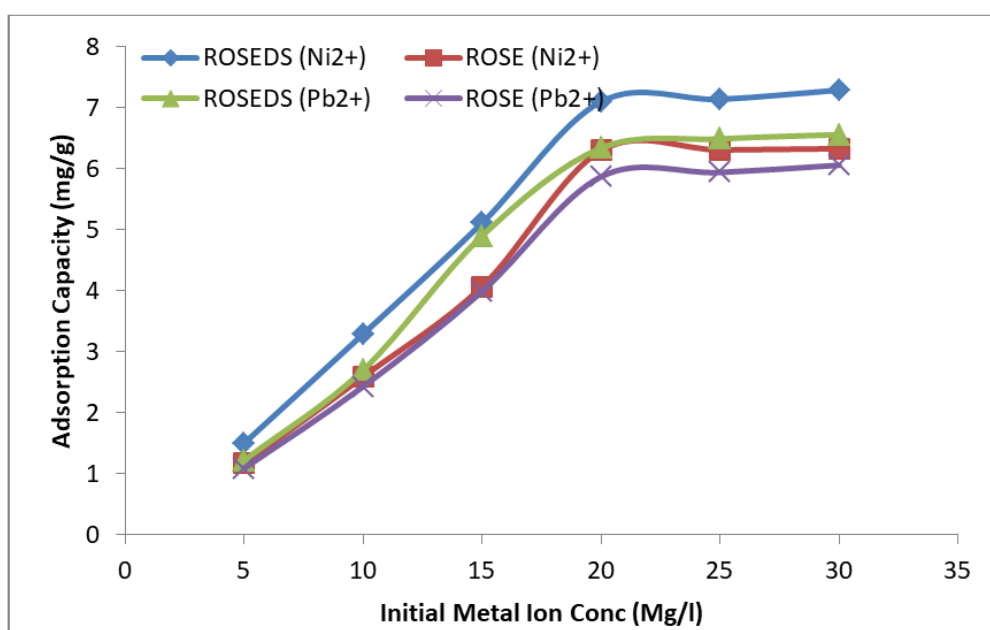
**Figure 2: Variation in metal ion concentrations (mg/l) on the percentage removal (%) of Ni<sup>2+</sup> and Pb<sup>2+</sup> ions using a fixed mass of ROSE and ROSEDS at their optimum pH values**



The results for adsorption capacities (mg/g) at varying metal ion concentrations (mg/l) of  $\text{Ni}^{2+}$  and  $\text{Pb}^{2+}$  ions using a fixed mass (0.54g) of (ROSE) and (ROSEDS) at their optimum pH values is presented in Figure 3.

From the results, at low metal ion concentrations, the adsorption capacity of ROSE for  $\text{Ni}^{2+}$  ion increased due to the excess binding sites on the adsorbent surface as compared to the few metal ions present in the solution (Lee 1996; Uzoukwu 2009). Increasing the metal ion concentration from (from 5.0 mg/l to 15.0 mg/l), while the number of binding sites on the adsorbent surface remained constant, led to an increase in the adsorption capacity of ROSE for  $\text{Ni}^{2+}$

from 1.2 mg/g to 4.07 mg/g, while that of ROSEDS for  $\text{Ni}^{2+}$  ion increased from 1.50 mg/g to 5.11 mg/g. At concentration of 20.0 mg/g, optimum adsorption of 6.30 mg/g was attained for ROSE, while the optimum adsorption of 7.10 mg/g was attained for ROSEDS. The optimum concentration is evidently the equivalent amount of exchangeable similar ions, indicating the attainment of saturation. Above this optimum concentration, an increase in the metal ion concentration did not yield any significant difference in the adsorption capacity of the adsorbents for the metal ion. This is because the limited adsorption sites on the surface of the adsorbents are occupied, leaving the excess metal ions in the solution not being adsorbed.



**Figure 3: Adsorption capacities (mg/g) at varying metal ion concentrations (mg/l) of  $\text{Ni}^{2+}$  and  $\text{Pb}^{2+}$  ions using a fixed mass of (ROSE) and (ROSEDS) at their optimum pH values**

A similar trend was observed for the adsorption of  $\text{Pb}^{2+}$  ions by the two adsorbents. The adsorption capacity of ROSE for  $\text{Pb}^{2+}$  ion increased from 1.09 mg/g to 3.99 mg/g as the metal ion concentration increased from 5.0 mg/g to 15.0 mg/g, while that of ROSEDS for  $\text{Pb}^{2+}$  ion increased from 1.22 mg/g to 4.90 mg/g. Optimum adsorption capacities were attained at 20.0 mg/l of the metal ion concentrations. A value of 5.87 mg/g was attained for  $\text{Pb}^{2+}$  ion using ROSE and 6.34 mg/g was attained using ROSEDS.

At low metal ion concentrations, the adsorption capacity increased due to the excess binding sites on the adsorbents surfaces, as compared to the amount of metal ion present in the solution; this in turn improves the removal of the adsorbates from the aqueous medium. Throughout the experiments, the uptake of metal ions by ROSE and ROSEDS was high, implying that the metal ions which are of higher

valence displaced the exchangeable similar ions which are of lower valence, preferentially. The degree of adsorption of the two metal ions by ROSE and ROSEDS is in the order:  $\text{Ni}^{2+} > \text{Pb}^{2+}$ . This observation is in agreement with the order of their ionic radii (0.72Å for  $\text{Ni}^{2+}$  and 1.21Å for  $\text{Pb}^{2+}$ ) (Lee 1996; Uzoukwu 2009). The above trend implies that smaller ionic radii could enhance the adsorbates uptake by ROSE and ROSEDS. Earlier research (Chukwu & Maduabuchi 2023) on the removal of heavy metal ions from aqueous medium noted that metals having larger ionic radii showed lower adsorption than the ones with smaller ionic radii. Therefore, the smaller the ionic radius, the easier it is for a metal ion to be adsorbed unto the adsorbent surface.

Results for the percentage removal (%) of  $\text{Ni}^{2+}$  and  $\text{Pb}^{2+}$  ions while varying the adsorbents (ROSE and ROSEDS) dosage at constant pH value is presented in Figure 4. The result of the effect of variation in

adsorbents dosage from 0.2 to 1.2 g/200 ml of ethyl acetate on the percentage removal of the metal ions and the adsorption capacity of the adsorbents showed that variation in adsorbent dosage is a crucial factor to be considered in the removal of heavy metals from aqueous medium.

The percentage removal of  $\text{Ni}^{2+}$  ion by ROSE increased from 51.6 % to 75.4 % as the mass of the adsorbent increased from 0.2 to 0.6g and attained optimum percentage removal of 89.9 % at 0.8g of adsorbent dose; while that of ROSEDS increased from 64.20 % to 91.30 % within the same range of adsorbent dosage, and attained optimum percentage removal of 97.90 % at 0.8g of the adsorbent dose. Thus, the optimum percentage removal of  $\text{Ni}^{2+}$  ion by ROSE and ROSEDS are 89.90 % and 97.90 %, respectively, at

0.8g of each adsorbent. Beyond the optimum dosage, further increase in the adsorbent dose did not yield any significant difference in the percentage removal of the adsorbates.

A similar trend was observed in the removal of  $\text{Pb}^{2+}$  ion by both adsorbents. The percentage adsorption of  $\text{Pb}^{2+}$  ion by ROSE increased from 48.6 % to 71.6 % as the adsorbent dosage increased from 0.2 to 0.6 g/ 200 ml of ethyl acetate, while that of ROSEDS increased from 57.3% to 81.5 % within the same range of adsorbent dosage. Optimum percentage removal of  $\text{Pb}^{2+}$  ion by ROSE and ROSEDS are 86.85 % and 90.80 %, respectively, at 0.8g of each adsorbent. Beyond the optimum dosage, further increase in the adsorbent dose did not yield a significant difference in the percentage removal of the adsorbates.

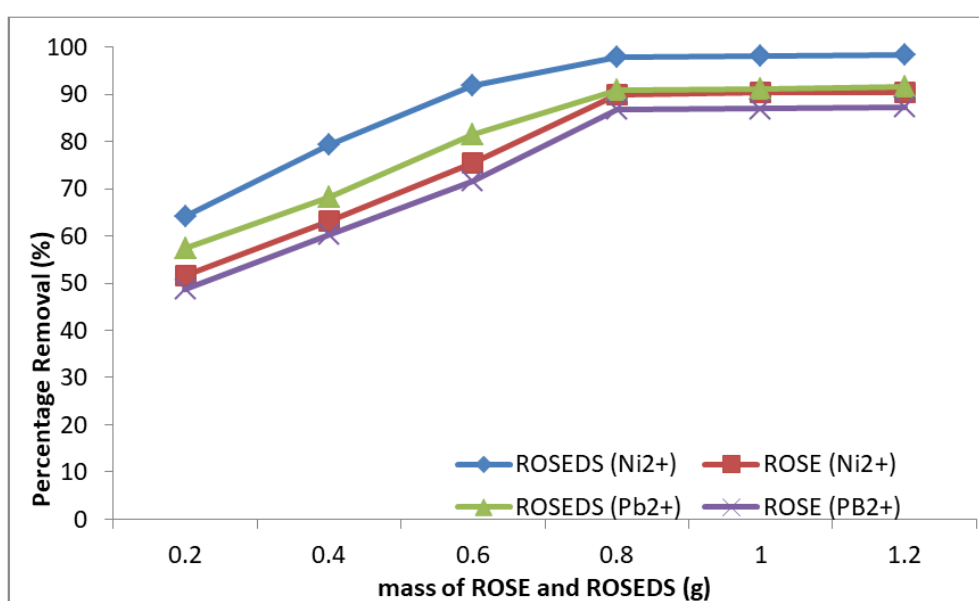


Figure 4: Percentage removal of  $\text{Ni}^{2+}$  and  $\text{Pb}^{2+}$  ions at while varying masses of ROSE) and ROSEDS at constant pH value

From the results of the adsorption capacities studies of both adsorbents for  $\text{Ni}^{2+}$  ion, the adsorbent dosage had an inverse relationship with the adsorption capacities of the adsorbents. That of ROSE decreased from 7.74 mg/g to 2.26 mg/g; while that of ROSEDS decreased from 9.63 to 2.05 mg/g as the adsorbent dosage increased from 0.2 to 1.2 g.

## CONCLUSION

The unmodified (ROSE) and 4-aminophenol modified red onion skin extract (ROSEDS) exhibited an appreciable potential for the removal of  $\text{Ni}^{2+}$  and  $\text{Pb}^{2+}$  ions, as the metal ion concentration and adsorbent dosage increased; and yielding optimum adsorption at 20.0 mg/l of the metal ion solution. While varying the metal ion concentration, optimum percentage removal of 86.1 % and 95.7% of  $\text{Ni}^{2+}$  ion by ROSE and ROSEDS, respectively were attained at 20.0 mg/l of  $\text{Ni}^{2+}$  ion. The optimum capacity of ROSE (6.3 mg/g)

and ROSEDS (7.10 mg/g) for  $\text{Ni}^{2+}$  ion were at the same optimum metal ion concentration at room temperature.

On the other hand, the optimum percentage removal of 79.3% and 85.6% of  $\text{Pb}^{2+}$  by ROSE and ROSEDS respectively, were attained at 20.0 mg/l of  $\text{Pb}^{2+}$  ion, while the adsorption capacities of ROSE and ROSEDS for  $\text{Pb}^{2+}$  ion are 5.87 mg/g and 6.34 mg/g, respectively at the same optimum concentration of the metal ion.

Variation in mass of ROSE and ROSEDS yielded optimum percentage removal of 89.9% and 97.9% of  $\text{Ni}^{2+}$  ion; while the adsorption capacities of ROSE and ROSEDS for  $\text{Ni}^{2+}$  ion varied inversely with the adsorbent dosage. That of  $\text{Pb}^{2+}$  ion gave optimum percentage removal of 86.8% and 90.8% ROSE and ROSEDS, respectively, at the optimum adsorbent dose of 0.8 g/ 200 ml of ethyl acetate. The adsorption capacities of ROSE and ROSEDS for  $\text{Pb}^{2+}$  ion varied

inversely with adsorbent dosage. The major removal mechanism was ion exchange between metal ions and positive ions.

### CONFLICT OF INTEREST

The authors (CUJ & AO) declare that there are conflicts of interest.

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