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Pb^{2+} Removal by Using Activated Coconut Waste Modified with Different Metal Oxide Nanoparticles

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ABSTRACT

Lead (Pb) is normally found in wastewater discharge from industrial manufacturing. The removal of lead ions (Pb^{2+}) is important to comply with the allowable wastewater discharge limit. To improve the removal of Pb^{2+} in wastewater, this study focused on investigating the effect of different metal oxides nanoparticles (MNPs), which are Fe_2O_3, NiO, and Al_2O_3 modified on activated coconut waste (ACW) and the effect of MNPs weight loading. The modification of different types of MNPs onto ACW is anticipated to investigate the effect of MNPs' characteristics towards ACW capability in removing the Pb^{2+}. To date, there is no work that reported on assessing the potential of MNPs modified ACW for Pb^{2+} removal, which would be a good benchmark for nanomaterials development in wastewater treatment application. Coconut waste was chemically activated to produce ACW, and it was modified with the MNPs to produce ACW/Fe_2O_3, ACW/NiO, and ACW/Al_2O_3 adsorbents. Batch adsorption method was employed at constant adsorption parameters to test the modified ACW/MNPs adsorbents performance. The result shows that the highest adsorption capacity of Pb^{2+} by using ACW/Fe_2O_3 adsorbents was 4.98 mg/g. As an increase in MNPs weight loading, the performance of the adsorbent was increased, where the removal efficiency calculated is more than 99%. From this study, it was found that the synthesis of ACW with three different MNPs has the potential to be an effective adsorbent in Pb^{2+} removal.

Keywords: *Activated Coconut Waste, Metal Oxides, Nanoparticles, Pb^{2+}, Adsorption, Wastewater.*

1. INTRODUCTION

Industrial wastewater normally contains organic, inorganic, and biological pollutants that are toxic and non-biodegradable [1]. In general, lead ions (Pb^{2+}) can be found in wastewater discharge by various sources such as battery manufacturing industries, television, printing, paints and dye, pigments, glass industries, photographic, electroplating, materials, and fuels [2][3][4]. Exposure to high concentration or dose of lead resulted in damaging the human organ and body system, especially the central nervous system, kidney and blood, which could cause death if over-exposed [5]. In addition, natural groundwater will also be contaminated, resulting in increased circulation of Pb^{2+} in soil, and water [5]. Referring to Environmental Quality Act 1974 (Sewage and Industrial Effluents) Regulations (Standard A and B), the allowable limit for Pb^{2+} in Standard A and B is 0.1 mg/L and 0.5 mg/L, respectively [6]. All industries are required

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to comply with the regulation. The Department of Environment (DOE) will regularly schedule the inspection to monitor the quality of wastewater.

Different methods and technologies were developed to overcome Pb^{2+} removal in wastewater, including chemical precipitation, ion exchange, electrolytic method, coagulation, ozonation, oxidation, reduction, membrane technology, and adsorption [7][8][9][10]. Among all, adsorption is a promising method in removing organic and inorganic metal in wastewater [7]. Adsorption is well-known due to various advantages such as low raw material costs, high performance and efficiency, flexibility, sludge-free production, cost efficiency and rigid discharge requirements [11][12][13]. Variety of adsorbents have been used as a precursor for Pb^{2+} removal, where various outcomes in terms of adsorption capacity, surface area and efficiency in Pb^{2+} removal were found. Wan et al. reported that activated coconut waste (ACW) is a great adsorbent in Pb^{2+} removal due to its physical and chemical properties, which obtained morphological structure like honey comb and it also consists of functional groups like hydroxyl (-OH), acetyl (-CH), and ester groups [14]. The adsorption capacity for non-modified coconut based activated carbon for Pb^{2+} is 94.35 mg/g [7] compared to other modified adsorbents that are capable of adsorbing more than 100 mg/g. Thus, to improve the adsorption capacity of the ACW, modification of the adsorbent needs to be done. The introduction of metal oxide nanoparticles could enhance the structure, surface area, and porosity of the adsorbent, while crafting an additional oxygen layer onto the structure [15].

Metal oxide nanoparticles (MNPs) exhibit diverse behaviors of bulk materials due to the large number of atoms at the material's surface or interface, which is applicable to be used in various applications such as environmental remediation, energy, and wastewater treatment. Several types of well-known metal-based nanostructure had been applied in adsorbent applications in removing heavy metal such as iron oxide (Fe_3O_4), titanium dioxide (TiO_2), nickel oxide (NiO), zinc oxide (ZnO) and aluminum oxide (Al_2O_3) [16][17]. MNPs have a large quantity to volume surface and have elevated paramagnetic layer characteristics. Owing to their comprehensive layer to volume ratio, transition of metal nanoparticles tends to accumulate, demonstrating strong super paramagnetic characteristics [16]. The main objective of this research work is to study the effects of different MNPs modified on ACW toward Pb^{2+} removal, while investigating their characteristics. Hence, in this paper, a modification of ACW by using different types of MNPs which are iron oxide (Fe_2O_3), nickel oxide (NiO) and aluminum oxide (Al_2O_3) is anticipated. The outcomes will address the research gap in assessing the effects of different types of MNPs' characteristics modified onto ACW for Pb^{2+} removal from wastewater. The MNPs were prepared by using a co-precipitation method prior to synthesis with the prepared ACW before being employed into batch adsorption of Pb^{2+}.

2. MATERIALS AND METHODS

2.1 Materials and Chemicals

In this work, the precursor used to produce the adsorbent is coconut waste, which was obtained from nearby a cafeteria. The chemicals used to modify the adsorbent are sodium hydroxide, NaOH (Merck), sulfuric acid, H_2SO_4 (Merck, 95-97%) ethyl alcohol, C_2H_5OH (Supelco, 96%) ammonia hydroxide, NH_4OH (Merck, 25%) iron (III) chloride hexahydrate, $FeCl_3.6\ H_2O$ (Merck, 99%), nickel (II) sulfate hexahydrate, $NiSO_4.6H_2O$ (Merck, 98%), and aluminum

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nitrate nonahydrate, $Al(NO_3)_3.9H_2O$ (Merck, 98.5%). Lead (II) nitrate, $Pb(NO_3)_2$ (Merck, 99.5%) was used to imitate the Pb^{2+} concentration in wastewater.

2.2 Preparation of Activated Coconut Waste (ACW)

Approximately 100 g of dried coconut waste was mixed with 200 mL of 0.1 M NaOH, and the slurry was heated at 80°C and stirred for 6 h. Next, the sample was mixed with 200 mL of 0.1 M of H_2SO_4. The mixture was heated at 80°C, while continuously stirred for 3 h. The mixture was then repeatedly washed with distilled water until the pH is close to 7. The sample was dried in an oven at 105°C for 24 h [18]. The dried activated coconut waste (ACW) was grounded and sieved to obtain particles size in the range of 250 μm prior to use for batch adsorption study.

2.3 Preparation of Metal Oxide Nanoparticles (MNPs)

The synthesis of MNPs was prepared by using a co-precipitation method. Firstly, a 10 mL of 2 M $FeCl_3.6\ H_2O$ was dissolved in a 10 mL of 2 M HCl. Then, a 2 mL of 1 M Na_2SO_3 was added to the mixture and was sonicated using an ultrasonic bath for 1 min. This was to enhance smaller and homogeneous particles. Upon mixing the liquids, the color of the solution observed was shifted from light yellow to red, showing the complex ions produced between Fe^{3+} and SO_3^{2-}. 80 mL of 0.85 M NH_3 was added after the solution changed the color. Black precipitate was developed, and it was allowed to fully crystallize for another 30 min. The precipitate was washed by magnetic decantation with distilled water until the pH of the suspension was less than 7.5. The suspension was dried in an oven under aeration (with air) at 105°C for 24 h until the color of the suspension slowly changed from black to reddish-brown. These steps were repeated for $NiSO_4.6H_2O$ and $Al(NO_3)_3.9H_2O$.

2.4 Modification of ACW with Different MNPs

To modify the adsorbent, a 100 mL of ethanol was poured into a beaker that consists of a 1 g of ACW. The mixture was stirred for 2 h at 80°C. Then, a 100 mL of aqueous solution containing Fe_2O_3 NPs was prepared at different loading weights and were mixed for 1 h (0.05, 0.25, 0.5, 0.75, 1.0 g) [14]. A 50 mL of NH_4OH solution was prepared, and the solution was poured slowly until the pH reached 7, while continuously stirred for 2 h. The modified ACW sample was filtered by using filter paper and washed with distilled water. Finally, the modified ACW sample was heated up in an oven at 50°C for 45 min under inert condition (N_2 gas flow) to produce ACW/ $FeCl_3.6H_2O$. The produced adsorbent was kept in a desiccator for later use. These procedures were repeated for NiO NPs and Al_2O_3 NPs at different loading weights.

2.5 Batch Adsorption Experiment

Adsorption experiment was performed in a batch reactor where Pb^{2+} removal was performed by adding modified ACW adsorbent (10 mg) at 150 rpm and 35 °C for 2 h in a shaking incubator. A 50 ml of $Pb(NO_3)_2$ solution with a concentration of 40 mg/L was used to simulate the Pb^{2+} concentration in wastewater. The pH values of the prepared solutions were adjusted using either 0.1 N of HCl or 0.1 N of NH_4OH. Adsorption parameters such as contact time, temperature, and MNPs loading were set to 30 min, 35 °C, and 1 g, respectively. Once completed, the solutions were centrifuged to evaluate the removal of Pb^{2+} at 1500 rpm. The removal of Pb^{2+} was analyzed by using ICP-OES. The removal efficiency, % R was calculated

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by using equation (1) and the amounts of metal ions adsorbed on ACW/MNPs adsorbent was calculated by using equation (2).

$$R(\%) = [Co - Ct / Co] \times 100\% \quad (1)$$

$$Qt = \frac{(Co - Ct)V}{m} \quad (2)$$

where, Co is initial metal ion concentration, Ct is residual metal ion concentration in solution at any time t (min), Qt (mg/g) is the adsorption capacity at any time t (min), V (L) is the volume of the solution, and m (g) is the mass of adsorbent used.

2.6 Thermal Gravimetric Analysis (TGA)

Thermal stability of ACW/MNPs adsorbent was analyzed by using thermal gravimetric analysis (TGA) with the presence of air and argon gases. The gas flow rate was set up at 50 mL/min at target temperature of 600 °C. The temperature ramping rate is 5 °C/min [14].

3. RESULTS AND DISCUSSION

To study the effects of MNPs weight loading on ACW adsorbent towards Pb^{2+} removal, different types of MNPs were used, which are Fe_2O_3, NiO, and Al_2O_3. The MNPs weight loading was varied in the range of 0.05, 0.25, 0.50, 0.75, and 1.0 g, respectively. The removal of Pb^{2+} was calculated based on the amount of Pb^{2+} adsorbed from the simulated wastewater. These MNPs were synthesized with 1g of ACW. The initial concentration of Pb^{2+} upon the adsorption is 100 mg/L. Detailed discussions are enclosed in the following sections.

3.1 Effect of Different Types of MNPs Modified on ACW toward Pb^{2+} Removal

The effects of different types of MNPs modified on ACW towards Pb^{2+} removal were analyzed and plotted in a graph as shown in Figure 1. At low MNPs weight loading (0.05 g), NiO showed good adsorption performance with adsorption capacity of 4.90 mg/g, whilst at high MNPs weight loading, Fe_2O_3 has greater performance with adsorption capacity of 4.98 mg/g. The performance of ACW modified with Al_2O_3 was also comparable with both Fe_2O_3 and NiO, since all adsorbents managed to adsorb more than 4.90 mg/g which is for about 97% of Pb^{2+} removal.

Fe_2O_3 has been extensively used to modify the adsorbent properties, due to its relatively elevated surface area, magnetic properties, and high oxygen functional groups [13]. Owing to their nano sizes and properties, these modified ACW/MNPs exhibit good performance in removing Pb^{2+} as well. Referring to findings from Mahdavi et al., it was reported that Fe_2O_3 performed better in biomedical applications contributing to its superparamagnetic behavior [16]. Superparamagnetic behavior is capable of adsorbing cationic charge heavy metal compared to normal paramagnetic behavior. However, in this work it was found that the performance of NiO and Al_2O_3 nanoparticles that were modified onto the ACW adsorbents were comparable to the performance of ACW/ Fe_2O_3.

Referring to Figure 1, the Pb^{2+} adsorption performance of these MNPs modified ACW was not following the trend as increasing in the MNPs weight loading. As for ACW/ Fe_2O_3, the MNPs weight loading at 1.00 g gave the best performance, which is 4.98 mg/g. However, the best

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performances for ACW/NiO and ACW/Al_2O_3 were found at MNPs weight loading lower than 1.00 g, which are 0.50 and 0.75 g MNPs weight loading with adsorption capacity of 4.95 mg/g and 0.75 g with adsorption capacity of 4.95 mg/g, respectively. As increasing the MNPs weight loading for ACW/NiO and ACW/Al_2O_3 up to 1.00g, the adsorption capacity was decreased for both samples. This finding might be contributed by a few factors such as the effect of crystallite size of the MNPs, the surface area of the adsorbent after being modified, and the available functional groups after modification. Instead of that, the possibility of inaccurate sample preparation was also taken into consideration in this matter since the adsorption capacity shown in Figure 1 is not much varied.

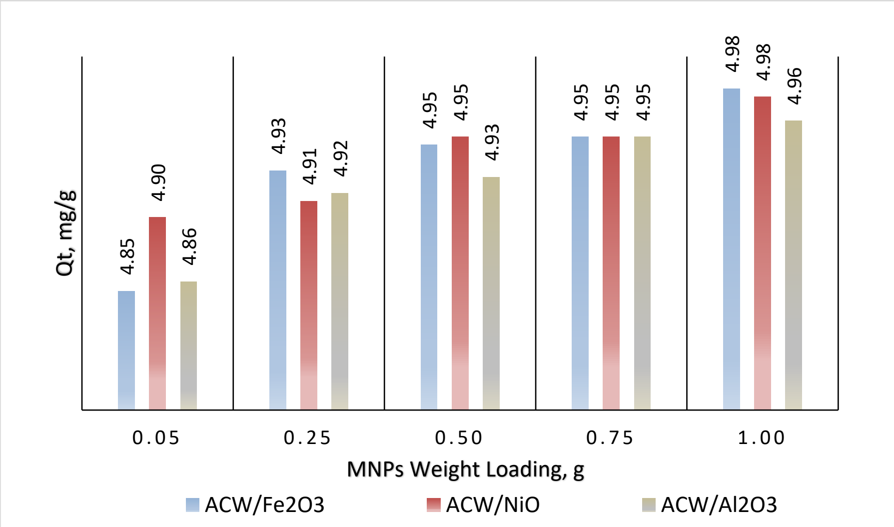

Figure 1: Effect of different types of MNPs modified on ACW toward Pb^{2+} removal

3.2 Effect of MNPs Weight Loadings towards Pb^{2+} Removal

The relationship of removal efficiency and total adsorption capacity for each modified ACW/MNPs adsorbent at different MNPs weight loadings has been plotted in graphs as illustrated in Figure 2. Figure 2a, 2b, and 2c illustrated graphs of ACW/Fe_2O_3, ACW/NiO, ACW/Al_2O_3 adsorbents, which show as an increase in removal efficiency, the adsorption capacity of Pb^{2+} was also increased. Increasing the MNPs weight loadings also resulted in the increase of Pb^{2+} adsorption onto the modified ACW/MNPs adsorbents. From Figure 2a, the minimum removal efficiency of Pb^{2+} is 97.08% at MNPs weight loading of 0.05 g and rapidly increases to 99.63% as the Fe_2O_3 weight loading to 1.0 g. As for ACW/NiO adsorbents in Figure 2b, at NiO weight loadings of 0.5 and 0.75 g, the performance for removal efficiency and adsorption capacity is the same (99% and 4.95 mg/g). The performance of the adsorbent starts to increase when the NiO weight loading was added up to 1.0 g. The trend for ACW/Al_2O_3 adsorbent is not much different with ACW/Fe_2O_3, however the performance of this adsorbent is less efficient compared to the ACW/Fe_2O_3 adsorbent.

The performance of removal efficiency and adsorption capacity of the modified ACW/MNPs adsorbent shows the same trend and increase as the MNPs weight loading increased. According to Dong et al., this occurrence is because the composite structure of ACW/MNPs has strong affinity toward Pb^{2+}, which the modification of these MNPs contribute to surface chemistry of additional available oxygen functional groups [19].

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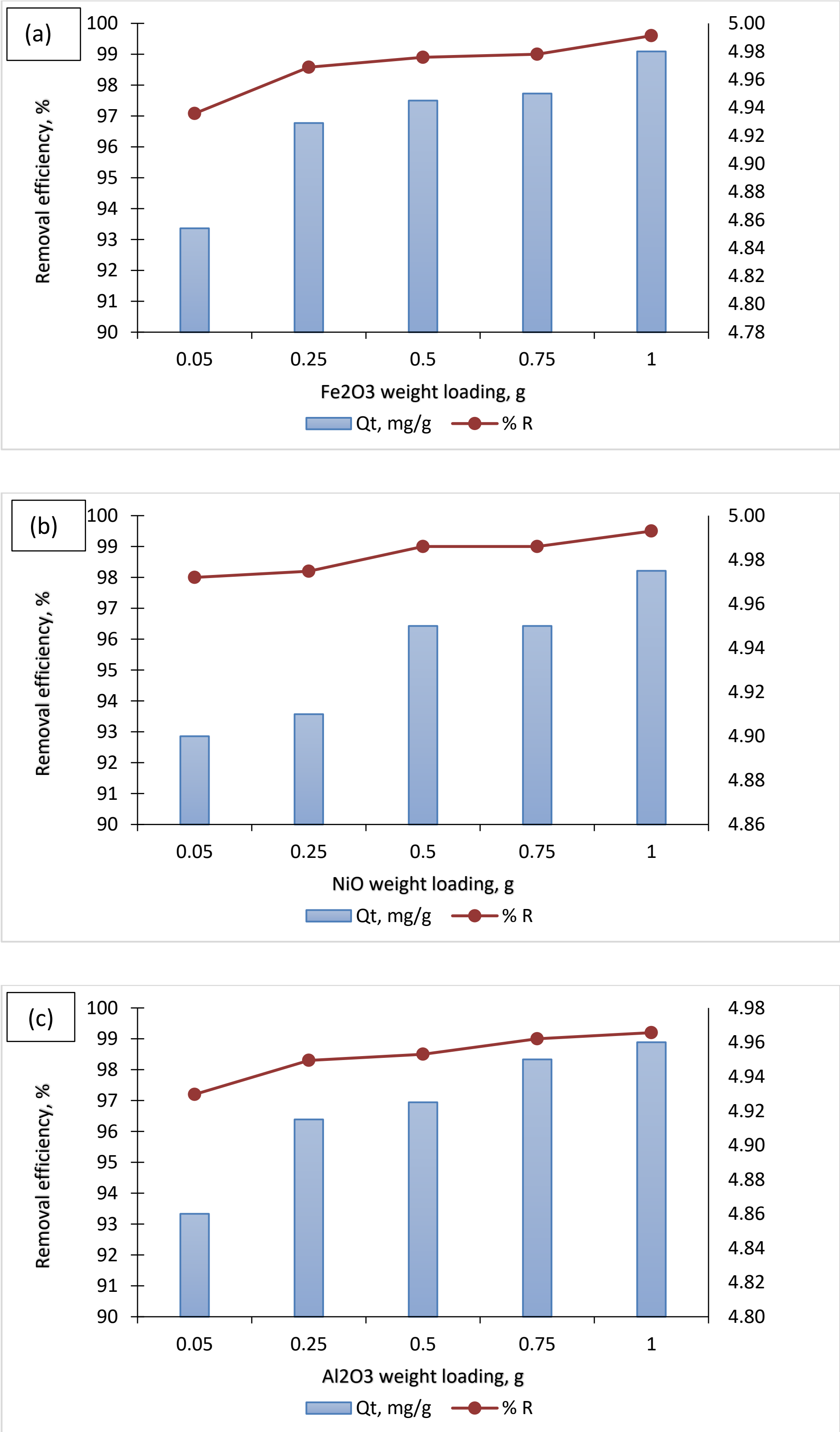

Figure 2: Effects of a) ACW/Fe_2O_3, b) ACW/NiO, and c) ACW/Al_2O_3 NPs adsorbents at different weight loadings toward Pb^{2+} removal

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The oxygen functional groups are responsible for the chemisorption of Pb^{2+} removal, where excess oxygen promotes the Pb^{2+} adsorption by increasing the activity of their neighboring carbon atoms. The van der Waals interaction that exists in the carbon layer between the hexagonally arrayed carbon atoms and the positively charged Pb^{2+} greatly assisted in the adsorption process [20]. Other than that, the electrostatic attraction between the positive cationic Pb^{2+} and the adsorbent surface charged negatively, as well as the electrostatic attraction between the pairs of electrons on the oxygen atoms of MNPs and the positive cationic Pb^{2+} are also promoting in the adsorption process [20]. The attraction that occurs is called a paramagnetic condition due to the different charge ions attracted to each other.

As observed in Figure 2, the highest performance of the modified ACW/MNPs adsorbent was shown by MNPs weight loading of 1.0 g for all ACW/Fe_2O_3, ACW/NiO, and ACW/Al_2O_3 adsorbents. As increasing the weight loading of MNPs, the surface area of the adsorbent will also increase. Due to the presence of internal pore, oxygen functional groups are capable to attract Pb^{2+} to be adsorbed at high MNPs weight loading because of the increment of available space for oxygen to attach onto the active site of MNPs. This is due to the nano size of MNPs further enhancing the pore structure of the adsorbent, where the MNPs were bound with the existing surface chemistry of ACW [21]. Therefore, as an increase in the number of adsorption sites available, will also increase the adsorption capacity of Pb^{2+}. This finding supports that the adsorption sites remain unsaturated until the maximum amount of Pb^{2+} concentration adsorbed onto the adsorbent, which is why the performance of the adsorbents are increasing as MNPs weight loading [22]. The combination of surface chemistry and physical properties of the modified ACW/MNPs adsorbent possess strong adsorption capability, where MNPs materials exhibit elevated catalytic properties, high surface area, and greater active sites to integrate with metallic species [23].

3.3 Thermal Stability of Modified ACW/MNPs Adsorbent

Thermogravimetric Analysis (TGA) is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere. In other words, TGA measures a sample's weight as it is heated or cooled in a furnace. In this study, the ACW/Fe_2O_3, ACW/NiO, and ACW/Al_2O_3 adsorbents were analyzed for thermal stability. The objective of this study is to record changes in mass due to dehydration, decomposition, and oxidation of a sample with time and temperature. Figure 3 shows the thermal stability curve for ACW/Fe_2O_3, ACW/NiO, and ACW/Al_2O_3 adsorbents.

The thermal stability curves of ACW/Fe_2O_3, ACW/NiO, and ACW/Al_2O_3 adsorbents were obtained at temperature of 50 – 600 °C (5 °C/min) and under nitrogen flow rate of 50 mL/min. The curves show an initial weight loss of ACW occurring below 100 °C due to the water loss altogether with moisture present in the samples. Although ACW was dried before the analysis, the total elimination of water was not completed because of the hydrophilic nature of the coconut fibers, which is present even as structurally bound water molecules [24]. The adsorbents were found to be stable at around 100 - 200 °C for ACW/Fe_2O_3, 80 - 285 °C for ACW/NiO, 90 - 250 °C for ACW/Al_2O_3, where an increase in degradation of weight was observed afterwards. Compared to ACW/Al_2O_3, the same trend was observed with a difference in degradation of weight, which is at 250 °C. This happened because the chemical composition of ACW/Al_2O_3 has a lower decomposition temperature compared to ACW/NiO and

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ACW/Fe_2O_3 [25]. The weight degradation of this range of temperature is due to the loss of volatile organic compounds [26]. From 300 °C and above, the sample showed little mass loss due to the decomposition of both cellulose and hemicelluloses in the fibers, which remained the MNPs [26]. The degradation of coconut fibers occurs might be due to the breaking bonds of lignin present in the fibers.

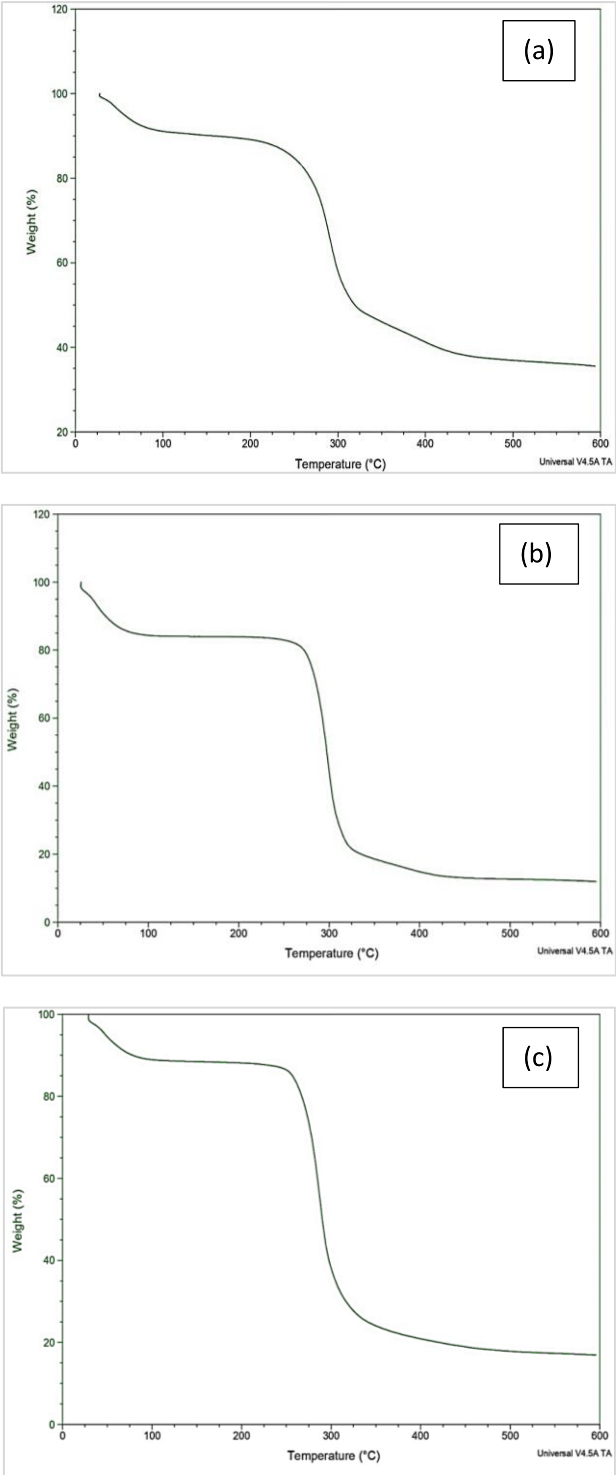

Figure 3: TGA curve for a) ACW/Fe_2O_3, b) ACW/NiO, and c) ACW/Al_2O_3 NPs adsorbents

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4. CONCLUSIONS

This study concluded that Iron (III) Oxide (Fe_2O_3), Nickel Oxide (NiO) and Aluminum Oxide (Al_3O_2) nanoparticles are potentially good in assisting the ACW adsorbent to remove Pb^{2+} in wastewater application. Overall, the results show an increase in MNPs weight loading varied from 0.05 to 1.0 g, increasing the performance of the modified ACW/MNPs adsorbents. The removal efficiency of Pb^{2+} for ACW/Fe_2O_3 adsorbent increases from 97.08 to 99.63%, for ACW/NiO adsorbent increases from 98 to 99.51%, and for ACW/Al_2O_3 adsorbent increases from 97.2 to 99.22%. The same trend was observed for total adsorption capacity of the modified ACW/MNPs adsorbents, where for ACW/Fe_2O_3, ACW/NiO, ACW/Al_2O_3 adsorbent increases from 4.854 to 4.98 mg/g, from 4.9 to 4.975 mg/g, and from 4.86 to 4.96 mg/g, respectively. The MNPs weight loadings suggest that highest weight loading could contribute to high performance of Pb^{2+} removal. ACW/Fe_2O_3, ACW/NiO, and ACW/Al_2O_3 adsorbents show promising results in Pb^{2+} removal. To improve the modified ACW/MNPs adsorbents, it is suggested to further analyze the physical and chemical characteristics of the adsorbent, while studying various synthesis parameters (i.e., pH, synthesis time, etc.).

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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