

Effect of pH and Particle Size for Lead and Nickel Uptake from Aqueous Solution using Cassava (*Manihot esculenta*) and yam (*Dioscorea alata*) Residual Biomasses Modified with Titanium Dioxide Nanoparticles

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Abstract

Background: Heavy metal water pollutants have received great attention due to its toxic effects on the environmental and health of human beings. Different techniques have been applied to remove heavy metal ions from aqueous solution including ion exchange, chemical precipitation and adsorption. **Objectives:** In this work, biosorption process was studied for nickel and lead ions uptake onto agricultural residual biomasses chemically modified with TiO₂. **Methods/Analysis:** The titanium dioxide nanoparticles were synthesized based on a green procedure using a leaf extract of lemongrass. Cassava and yam peels biomasses (CP and YP) were prepared and loaded with these nanoparticles through an organic solvent. The resulting biosorbents (CP-TiO₂ and YP-TiO₂) were characterized by FT-IR and SEM analysis in order to identify functional groups and morphology. The effect of pH and particle size on removal yield was evaluated by carrying out batch adsorption experiments at room temperature and fixed biosorbent dosage. **Findings:** It was observed characteristic peaks of titanium dioxide in FT-IR spectra of biosorbents confirming its successful synthesis. The carboxyl and hydroxyl groups were also identified, which can easily bind with metal ions to remove them from the solution. The surface of biosorbents showed a non-porous and heterogeneous morphology. The solution pH=6 was selected as suitable value according to adsorption result and point of zero net charge. The particle size did not significantly affect adsorption performance of biomaterials. The removal yields were 99.84% and 99.85% for Pb (II) using CP-TiO₂ and YP-TiO₂, respectively. For Ni (II), the removal yields were 81.51% and 86.66% using CP-TiO₂ and YP-TiO₂ biosorbents. **Novelty/Improvement:** These results suggested that agricultural wastes, such as cassava and lemon peels, can be used to prepare biosorbents with high adsorption efficiency and its modification with nanoparticles allows attracting greater amount of heavy metal ions increasing removal yields.

Keywords: Biotechnology, Biomass, Biosorption, Nanomaterials

1. Introduction

Heavy metals water pollutants are considered a severe environmental concern worldwide due to their non-bioaccumulation nature, high toxicity, carcinogenicity, prevalence, existence and persistence in the environment^{1,2}. Lead is one of the most common heavy metals in industrial waste waters such as the production of dyes, paint coatings, glass and batteries, and it is known to be toxic for humans and plants³. The exposure of humans to this heavy metal causes severe acute poisoning, learning disabilities for children, damage to organs and immune system disorders⁴. Nickel is one of the most commonly used heavy metals in metal processing and its exposure can produce anemia, diarrhea, encephalopathy, hepatitis, lung and kidney damage, gastrointestinal distress, pulmonary fibrosis, renal edema, skin dermatitis and central nervous system dysfunction^{5,6}. Conventional methods for nickel and lead uptake exhibit disadvantages such as high operational cost, energy requirements and generation of toxic wastes⁷. Alternative solutions have become of eminent importance, searching for techniques that aid in the removal of these contaminants⁸. Biosorption seems to be a promising new technique due to its low costs, easy operation, high efficiency and affinity of biomaterials for heavy metals⁹.

Several materials from agricultural wastes have been tested to adsorb heavy metal ions because of its availability, low cost and efficiency¹⁰, which include: corn cob, orange peels, sugarcane bagasse, lemon peels, cassava peels and cucumber peels^{2,11-15}. In addition, great efforts have been made to develop novel biosorbents by chemically modifying biomass with compounds such as citric acid, calcium chloride and nanoparticles^{16,17}. The biosorption of metal ions using biomaterials and nanoparticles from magnetic particles (e.g. titanium dioxide, cerium oxide, zinc oxide) offers many advantages related to nanotechnology application as reduction of mass transport resistance due to the high surface area and adsorption capacity of nanomaterials¹⁸. In this work, the adsorption process of Ni (II) and Pb (II) was carried out using cassava and yam peels biomasses chemically modified with TiO₂ nanoparticles. The synthesized biosorbents were characterized by SEM and FT-IR analyses to identify morphology and main functional groups associated to heavy metals uptake. The effect of pH and particle

sizes was evaluated in order to select suitable operating parameters value. Additionally, the removal yield was calculated to analyze the improvements of adsorption process after loading titanium dioxide nanoparticles onto biomasses.

2. Material and Methods

2.1 Synthesis of TiO₂ Nanoparticles

The TiO₂ nanoparticles were obtained by green synthesis using a leaf extract of lemongrass, which reduced titanium (IV) isopropoxide as pointed out¹⁹. The lemongrass was washed thoroughly with water, dried and milled. Then, 100 g of milled lemongrass was added to 500 mL of distilled water on a beaker placed on a hot plate at 86°C. After 30 minutes, the mixture was filtrated repeatedly to obtain 50 mL of extract. This extract (15 mL) was mixed with 85 mL of Ti[OCH(CH₃)₂]₄ solution and stirred during 24 hours. The nanoparticles were separated from the solution by centrifugation at 5000 rpm for 15 minutes. Then, they were washed with distillate water and ethanol and dried at room temperature as reported by²⁰. The titanium dioxide sample was heated in a muffle at 550°C for 3 hours in order to obtain a crystalline structure with high anatase composition, hence; the heating rate was fixed in 1 °C per 3 minutes.

2.2 Preparation of Biosorbents

Cassava (*Manihot esculenta*) and yam (*Dioscorea rotundata*) peels were purchased from a local market and used as sources of biomass to synthesize biosorbents. The peels were cut into small pieces and washed with water and ethanol to remove impurities that can affect adsorption process. The biomass was dried for 24 hours at 80°C, grounded and sieve-meshed to 0.355, 0.5 and 1 mm^{12,13}. For loading TiO₂ nanoparticles, 0.5 g biomass was mixed with Dimethyl Sulfoxide (DMSO) solution and continuously stirred at 120 rpm. The Ultraturrax equipment was used to properly dissolve the biomass and avoid the formation of clots by ultrasound technique. This suspension was added to 3 mL of Tetra Ethyl-O-Silicate (TEOS) used to induce hydrolysis and condensation of molecules. Afterward, the nanoparticles (0.5 g) were added and centrifuged at 3000 rpm. The resulting biosorbents (YP-TiO₂ and CP-TiO₂) were washed with ethanol and dried in an oven²¹.

2.3 Characterization Techniques

The biomasses before and after loading TiO₂ nanoparticles were characterized by Fourier Transform Infrared Spectroscopy (FT-IR) technique in order to identify main functional groups associated to adsorption interaction with heavy metal ions. Scanning Electron Microscopy (SEM) was also performed to study morphology of synthesized biosorbents.

2.4 Determination of Point of Zero Charge

The point of zero charge is the pH value at which a particle is electro-kinetically uncharged²². After adsorbing H⁺ or OH⁻ ions, biomaterials changes the net charge of its surface and the pH value that produces this phenomena is called as point of zero net protonic charge or isoelectric point, which mainly depends on the nature of solids²³.

To determine the point of zero charge (pH_{PZC}), the pH of 50 mL of distillate water was adjusted to 3, 4, 5 and 6 using HCl and NaOH solution. Then, 0.5 g of biomaterials were added and continuously stirred at 150 rpm for 46 hours. The measurement of final solution pH was required to construct a plot of pH variations. The pH_{PZC} of biomaterials corresponds to pH range at which there are not changes in final pH values²⁴.

2.5 Batch Adsorption Experiments

Nickel Sulfate (NiSO₄) and lead chloride (PbCl₂) were purchased from Sigma Aldrich and dissolved in deionized water to obtained stock solutions at 100 ppm. The

effect of pH on lead and nickel uptake was evaluated by adjusting pH solution in 2, 4 and 6 through NaOH and HCl. The batch experiments were carried out in a flask placed on a Shaking incubator IN-666. The biosorbent dosage was fixed in 5 g/L and different particle sizes (0.355, 0.5 and 1 mm) were considered to determine suitable conditions for performing further experiments. After adsorption process, the resulting solutions were vacuum filtrated and a sample of eluent was taken. The remaining heavy metal concentration was determined using absorption spectrophotometry technique¹⁴. The removal yield was calculated by Equation 1.

$$\text{Removable yield}(\%) = \frac{(C_o - C_e)}{C_o} \cdot 100\% \quad (1)$$

Where C_o (mg/L) and C_e (mg/L) are the initial and remaining concentrations of heavy metal ions, respectively.

3. Results and Discussion

3.1 Characterization of Biosorbent

Figure 1 shows FT-IR spectrum of both biomass and chemically modified biosorbents. The presence of absorption bands characteristic of titanium dioxide indicated the successful synthesis of biosorbents. It was found a sharp peak at 1650 cm⁻¹ attributed to stretching vibrations of hydroxyl groups bonded to Titanium atoms (Ti-O). The bands around 1336 and 940 cm⁻¹ are assigned to R-COO-Ti. The characteristic peaks at 1456, 1455, 1045 and 800 cm⁻¹ correspond to Ti-O-N,

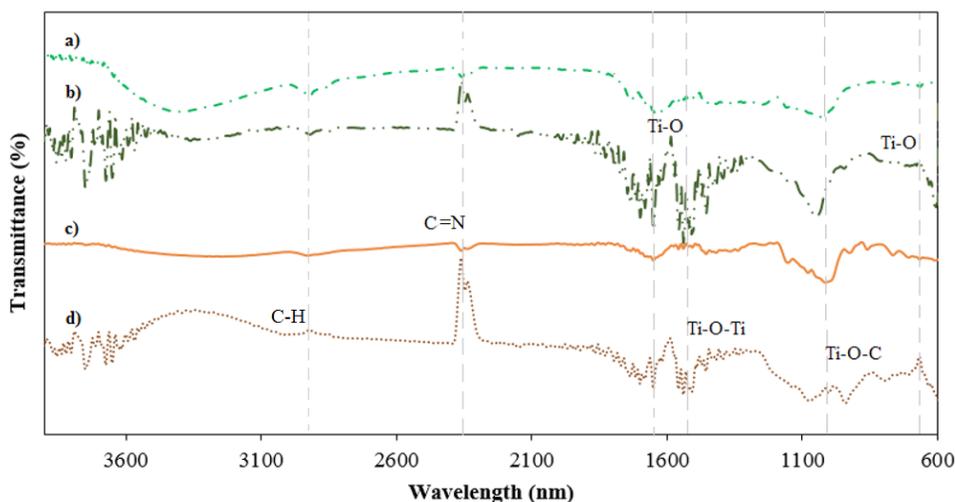


Figure 1. FT-IR spectrum for: (a) YP, (b) YP-TiO₂, (c) CP and (d) CP-TiO₂ biosorbents.

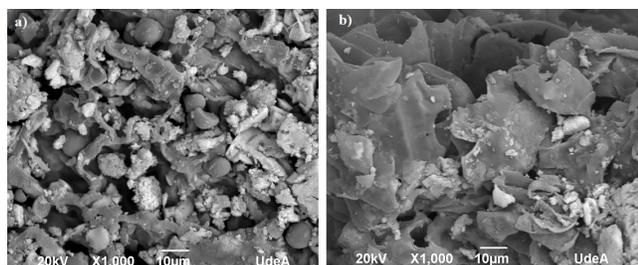


Figure 2. SEM micrograph of: (a) CP-TiO₂ and (b) YP-TiO₂ biosorbents.

Ti-O-Ti, Ti-O-C and Ti-O bonds²⁵⁻²⁷. It has been reported that carboxyl and hydroxyl groups can easily bind with metal ions to remove them from the solution^{15,28}, which are observed in all biosorbents spectrum. The band around 1000 and 3650 cm⁻¹ are assigned to carboxylate and -OH groups of cellulosic materials^{29,30}. In addition, C=N stretching vibrations of nitrile groups and C-H of alkanes and methylene groups were identified at 2300 and 2850 cm⁻¹, respectively³¹. The presence of these groups suggests an improvement in adsorption process of nickel and lead onto YP-TiO₂ and CP-TiO₂ biosorbents^{32,33}.

The micrographs of biomaterials chemically modified with TiO₂ nanoparticles at 1000x magnification are shown in Figure 2. It was found non-porous and heterogeneous characteristics in both biosorbent surface, which are similar to that reported by Mohd-Asharuddin³⁴ on SEM analysis on cassava and yam peels. In addition, the presence of TiO₂ nanoparticles generates an irregular morphology due to the agglomeration of primary particles²⁷.

3.2 Determination of Point of Zero Charge

It is well known that solutions at low pH exhibit more H⁺ cations, which is not accurate to attract heavy metal ions. The negative charges at higher pH makes possible the preferential interchange of cations improving the retention of heavy metal ions onto biomass. Hence, pH values above and below of pH_{PZC} exhibit negative and positive net charge, respectively³⁵. As is observed from Figures 3-4, the value of pH at zero charge is 4.65 and 5.92 for cassava and yam peels biomasses, respectively. These results suggested that pH = 6 could be favourable for performing adsorption process instead of pH = 2 and 4, because of the high attraction of cations (heavy metal ions) at pH values above pH_{PZC}.

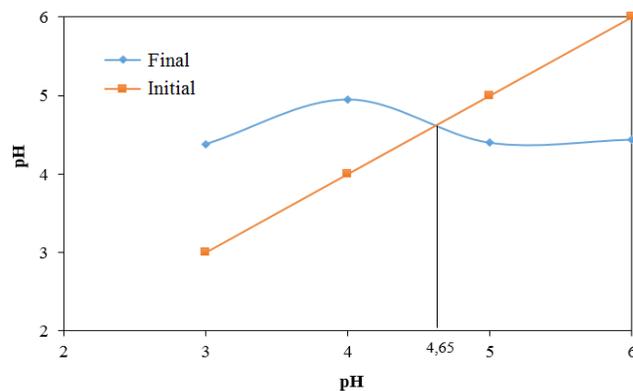


Figure 3. Point of zero charge for cassava peel biomass.

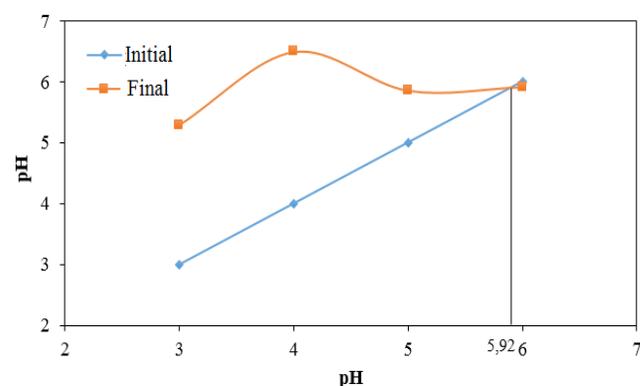


Figure 4. Point of zero charge for yam peel biomass.

3.3 Adsorption Experiments

3.3.1 Effect of pH and Particle Size

The solution pH and biosorbent particle sizes are key factors that should be considered in adsorption process since they substantially affect the mechanism of metal ions uptake³¹. Three pH and particle size values were selected to carry out batch experiments and these results were used to determine suitable operating conditions based on removal yields. The effects of both factors on removal yield are shown in Figures 5-6 for lead and nickel ions, respectively. As is observed in Figure 5, the removal yield increased steadily by increasing pH from 2 to 6, which is attributed to the competition between Pb (II) ions and H⁺ ions for the adsorption sites and the protonation of functional groups in biosorbent surface at lower pH reducing the amount of lead uptake. In addition, this effect could be explained according to the pH at point zero charge of the biosorbent as pointed out³⁶. The surface charges of the adsorbent are positive at pH < pH_{PZC} which

lead to an electrostatic repulsion with Pb (II). Similar results were reported by¹² using orange peels as biosorbent.

As shown in Figure 6, both biomasses exhibited similar performance for removing nickel ions at pH = 4 and 6. In³⁷ reported limitations of the sorption capacity of Ni (II) as electrostatic repulsion increased at lower pH. Furthermore, the effect of particle size is not significant on removal yields. The average removal yield was 73% at pH = 4 and 74.5% at pH = 6 using cassava peels biomass. For yam peels biomass, the average removal yield was calculated in 73 and 75.3 % at pH = 4 and 6, respectively. Hence, pH = 6 and particle size=0.355 mm were selected as suitable operating conditions for further experiments and Table 1 summarizes removal yields obtained at this conditions. Additionally, it can be observed that yam peels presented better results for nickel and lead uptake than cassava peels biomass, which is attributed to the greater content of lignin in yam peels increasing the presence of hydroxyl and carboxyl groups.

Figure 7 shows the removal yield of nickel and lead using biomasses and its modifications with TiO₂ nanoparticles. These results indicated that chemical modification with dioxide titanium nanoparticles enhances

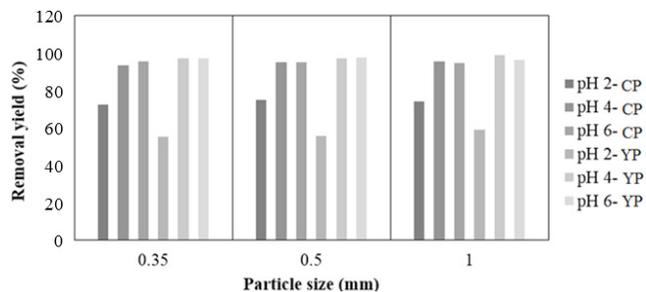


Figure 5. Effect of particle size on removal yield of lead using yam and cassava peels at pH=2, 4 and 6.

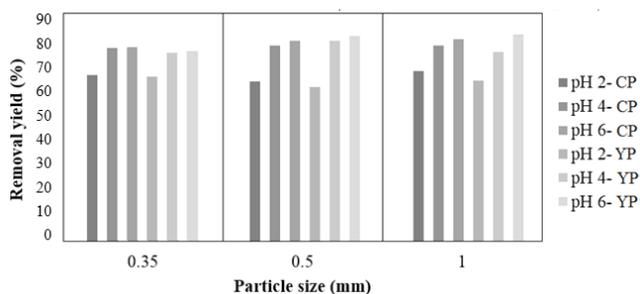


Figure 6. Effect of particle size on removal yield of nickel using yam and cassava peels at pH=2, 4 and 6.

Table 1. Removal yields of Ni (II) and Pb (II) using CP and YP biomass

Biomass	Removal yield (%)	
	Ni (II)	Pb (II)
CP	72.90%	97.28%
YP	71.38%	95.55%

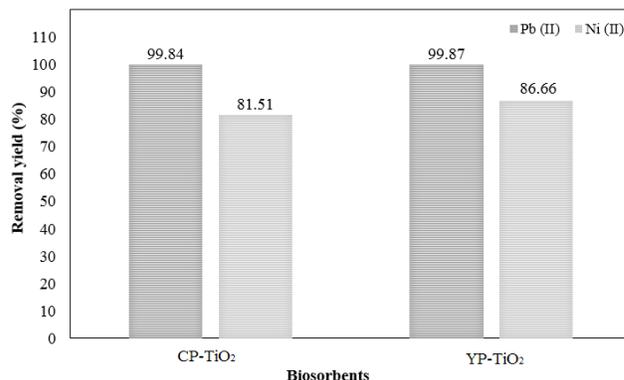


Figure 7. Removal yield of lead and nickel using chemically modified biosorbents at pH=6.

the performance of cassava and yam peels to Ni (II) and Pb (II) uptake. The Pb (II) ions removal yields reached were 99.84 and 99.87% using CP-TiO₂ and YP-TiO₂, respectively. For Ni (II) ions, the removal yield values were 81.51 and 86.66 % using CP-TiO₂ and YP-TiO₂, respectively. Similar results were obtained by Li et al., who claimed that biomaterials modified with TiO₂ nanoparticles are more efficient to heavy metals uptake from waste water, especially lead. On the other hand¹¹ reported removal yields of Pb (II) ions adsorption onto amorphous nano-aluminophosphate ranged 40-70%²⁵ also obtained removal yields above 90% using TiO₂ nanoparticles confirming the high adsorption capacity of this nanomaterial to adsorb heavy metal water pollutants. In³⁸ studied nickel and lead adsorption using cassava peels biomass and obtained the highest removal yields of 75.69% for Ni (II) and 97.5% for Pb (II) at pH = 6 and particle size = 0.5 mm. Other work exhibited Pb (II) removal yields of 70 and 96% using cassava and yam peels, respectively³³.

4. Conclusions

Thus study was to prepare biosorbents from cassava and yam peels and TiO₂ nanoparticles and to apply them for removing Pb (II) and Ni (II) ions from aqueous solution.

The FT-IR analysis revealed the presence of hydroxyl, carboxyl and R-COO-Ti groups. Adsorption bands characteristic of titanium dioxide nanoparticles were also observed, which confirmed the successful synthesis of biosorbents. The SEM analysis showed an irregular structure similar to that reported for TiO₂ nanoparticles. The pH was identified as the factor that most contributes to adsorption process. According to removal yield results and point of zero net charge, pH = 6 was selected as suitable value for carrying out further experiments. In addition, it was calculated removal yields of 99.84% and 99.85% for Pb (II) using CP-TiO₂ and YP-TiO₂, respectively. For Ni (II), the removal yields were 81.51% and 86.66% using CP-TiO₂ and YP-TiO₂ biosorbents. Hence, it can be concluded that chemical modification with titanium dioxide nanoparticles offers an attractive alternative for developing novel biosorbents with high adsorption efficiency.

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