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ORIGINAL RESEARCH PAPER

Removal of Heavy Metals (Lead and Nickel) from Water Sources by Adsorption of Activated Alumina

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Abstract: Textile industries are the major industries which mainly use different dyes. In these industries it is estimated that 15 % of dyes are lost in wastewater during manufacturing and dying process. In this study, the photocatalytic degradation of Yellow Acid-36 dye in the presence of UV irradiation intensity has been investigated. In this experiment TiO_2 P-25 was used as catalyst and coated on the marble surface. The influence of various operation parameters, such as the initial concentration of dye, pH, time of contact, catalyst dosage and UV irradiation intensity on the degradation process was examined. The results indicated that the optimal decolorization conditions were attained with 100 g m^{-2} of catalyst at pH 3, an irradiation time of 105 min, UV irradiation intensity 30 W and 5 mg L^{-1} initial concentration of dye. The process resulted in 97.6% degradation of Yellow Acid 36. It is revealed that this method was effective in degradation of Yellow Acid 36.
Keywords: Adsorption, Activated alumina, Heavy metals, Lead, Nickel, Water treatment

1. Introduction

Heavy metals are a group of metals with a specific gravity of more than 5 g/cm^3 or an atomic mass of more than 60 to 200 (Chen et al. 2019; Arjaghi et al. 2021). Heavy metal ions (HMIs) like zinc (Zn), lead (Pb), mercury (Hg), silver (Ag), arsenic (As), cadmium (Cd), chromium (Cr), and nickel (Ni) are a class of inorganic pollutants introduced into water bodies through untreated waste effluents majorly from industries such as mining, fertilizers, batteries, pesticides, refining, tanneries, and paper and are an increasingly critical threat to healthy ecosystems (Schaider et al. 2014; Sharma and Bhattacharya 2017). HMIs widely exist in all kinds of environments which could accumulate in the human body to a high concentration through the food chain from contaminated water (Li et al 2018; Rajaei et al., 2020). HMIs may be hazardous and life-threatening for humans, animals, and aquatic bodies after exposure to even low concentrations. Heavy metals such as Pb and Ni are non-degradable and tend to accumulate in living organisms, causing several diseases and disorders for humans, other living organisms and the natural ecosystem. Pb pollution sources are industries such as mining, steel, automobile, batteries and paints. Pollutants arising from increasing industrialization are listed as Pb pollution sources. Ni sources are effluents of silver refineries, electroplating and storage battery industries. Contamination to Pb and Ni compounds may cause dermatitis, kidney damage, muscle weakness, myocarditis, encephalopathy, pulmonary fibrosis, cancer, headache, dizziness, nausea and vomiting, chest pain and rapid respiration (Lakherwal 2014; Khalili Arjaghi et al. 2020). According to their long-term persistence, toxic nature, and non-biodegradable and bioaccumulation behavior, their presence in wastewater effluents and subsequent release to the environment may result in detrimental effects (Sibanda et al. 2015; Whitehead et al. 2018). Consequently, HMIs elimination from contaminated wastewater effluents before being discharged to water bodies is a dominant concern.

Several methods of heavy metals removal have been reviewed such as chemical precipitation, reverse osmosis, ion exchange, ultrafiltration, electrodialysis, coagulation, nanofiltration, flocculation and floatation. However, these methods have several disadvantages such as high reagent requirement, generation of toxic sludge and unpredictable metal ion removal (Khulbe and Matsuura, 2017; Nouri Dodaran, 2019). Some environmental friendly methods such as biosorption have a good performance as a low-cost internal method for water and wastewater treatment (Gooran Ourimi and Nezhadnaderi 2020). Typically, Heavy metals is used for the removal of water impurities causing the smell and color of the water, metals, and anions. The Adsorption process seems to be very simple, effective, economical and versatile which could be the most preferred method for the

removal of toxic contaminants (Lakherwal 2014; Omrani and Fataei, 2018). The adsorption process is a significant technique due to its wide applications, such as ease of operation, economic feasibility, wide availability and simplicity of design (Malik et al. 2016).

One of the inorganic sorbents used for removing the components present in water is activated alumina. It is a mixture of amorphous aluminum oxides obtained by dehydrogenation of aluminum hydroxide. Activated alumina is a granular form of aluminum oxide (Al_2O_3) with an adsorption level of 200-300 m^2/g. Activated alumina could remove arsenic from drinking water and tended to adsorb many negatively charged components such as textile dye (Wasti and Awan 2016). Because of its crystalline structure, it is highly selective relative to specific ions. The removal mechanism includes the exchange of hydroxyl ions for soluble species (Liu et al. 2019). Activated alumina is much less likely to be used for water treatment than activated charcoal; however, it is used to remove inorganic contaminants such as fluorides, arsenic, selenium, and silicates (Szatylowicz and Skoczko 2018).

It was initially assumed that anion removal by activated alumina was a pure ion exchange process. But now it is found that the process is more complex than previously thought, and at least a part of the process is adsorption in the case of some anions, which can be simulated using the Langmuir isotherm. According to the studies (Karagedov 2019), the selectivity of most activated alumina represented below:

Hydroxide (OH^-) > Dihydrogen Arsenate ($H_2AsO_4^-$) > Flouride (F^-) > Sulfate (SO_4^{-2}) > Bicarbonate (HCO_3^-) > Chloride (Cl^-)

In general, it is tried to use different types of adsorbents due to their adsorption and ion exchange properties in the removal of heavy metals, because of advantages such as inexpensiveness, high availability, fast adsorption and high adsorption capacity and reuse have led to the development of adsorbents for the removal of heavy metals (Sun et al. 2020). Accordingly, this study investigates the efficiency of activated alumina in removing Pb and Ni from water sources.

2. Materials and Methods

This study consisted of determining the effect of activated alumina on the removal of Pb and Ni in water sources and its efficiency to several variables. The experiment was conducted at a laboratory scale using model solutions. The removal efficiency of heavy metals has been determined due to variable Pb and Ni concentrations, pH, contact time and adsorbent dose. Initial laboratory tests were performed in 12 volume of laboratory solutions of different Pb and Ni concentration range. Then 16 samples of Pb and Ni solutions were tested by different pH of solutions. The third stage of the laboratory test process was applied by 8 samples in variable adsorbent dose. The final tests were applied by 10 samples of different contact times.

Standard solutions of Pb and Ni metals were prepared by dissolving certain amounts of Pb and Ni nitrate salts by adding a few drops of nitric acid and then deionized distilled water was prepared. The concentration of standard solutions of 1000 mg/l (ppm) was considered as the stock solution and was used to prepare a solution with a lower concentration. Also, nitrate salts and pH adjustment solutions are of high purity (made by Merck Company) were materials used in experimental work.

Physical and chemical properties of activated alumina (Merck, Germany) as a mineral adsorbent were determined. Meshes with dimensions of 0.2 to 2 mm have been selected and while homogenizing the particles, washed several times with distilled water to remove dust and other contaminants, then dried at 105 °C to achieve a constant weight. Chemical analysis of activated alumina applied in this study has been analyzed which is represented in Table 1. As observed, most of this mineral is composed of Al_2O_3 and a small percentage of this mineral contains sodium, silicon and iron oxides.

Table 1 Chemical analysis of activated alumina

Components	Content
92%	Al_2O_3
0.9%	Na_2O
0.09 %	SiO_2
0.08 %	Fe_2O_3

The batch method has been used in all experiments. A solution with a volume of 50 ml and a certain concentration of Pb and Ni metals at a constant pH was added to each flask and then different content values of activated alumina were added to them. After mixing to reach equilibrium time, the suspension was measured by filter paper with a smooth aqueous band and the concentration of metals in the filtration solution by atomic absorption (AA) method. Pb and Ni were measured by standard method 3113 B electro-thermal atomic absorption spectrometric method at 350 nm and 3113 B electrochemical atomic absorption spectrometric method at 350 nm, respectively. Accordingly, the effect of the disturbing parameters namely pH, contact time, metal concentration and adsorbent dose were investigated by maintaining all the parameters constant and changing only one of them.

In all tests, the conditions such as 50 ml volume of solution in each flask, 0.5 mm mesh size of particles, pH equal to 9, the temperature of 27 ± 1°C, mixing speed of 120 rpm, contact time of 30 min and metal concentration of 20 mg/l were constant values and in each test, one of the parameters supposed as variable. Table 2 shows the test methods used for the studied parameters.

Table 2 Experimental methods used for the studied parameters

Test	Unit	Test method	Instrument Model	Manufacture
Pb and Ni Concentration	mg/l	Atomic Absorption Spectrophotometry	PG990	PG Instruments, England
pH	-	pH Measurement	F-11 pH Meter	Horiba, Japan
Activated Alumina Dose	g/l	Weighing	BP210 D Analytical Balance	Sartorious, Switzerland
Contact Time	min	Timing	-	-

The following formula has been used to calculate the adsorption rate of each metal ion by the adsorbent (Ayawei et al. 2017):

$q_e = (C_o - C_e)\ V/m$ (1)

Where:

q_e: The amount of metal adsorbed per adsorbent weight (mg/g)

C_o: initial concentration of metal in the solution (mg/l)

C_e: Equilibrium concentration of metal in solution (mg/l)

V: volume of solution (ml)

m: adsorbent mass (gr)

The adsorption rate for a wide range of metal concentrations can be described by adsorption isotherms. Adsorption isotherms are described in both quantitative and qualitative ways. In the quantitative description, the most important isotherms are Langmuir and Freundlich isotherms, in which isotherms are determined by the ability and adsorption capacity of metal ions by examining the correlation and constant of isotherms (Chen 2015). In the first method, if q_e is the content of adsorbed metal per unit weight of adsorbent and C_e is the equilibrium concentration of the metal in the solution, the Freundlich adsorption isotherm is according to the following equations (Boparai et al 2011):

$$q_e = K_f C_e^{1/n} \quad (2)$$

$$\log q_e = \log K_f + {}^{1}/_{n}\ \log C_e \quad (3)$$

Where:

K_f and n are the Freundlich adsorption isotherm constants representing the adsorption capacity and adsorption energy, respectively.

The Langmuir isotherm equation used for equilibrium adsorption is as followed (Tripathi et al 2019):

$$C_e/q_e = {}^{1}/_{Q_o}\ b + {}^{C_e}/_{Q_o} \quad (4)$$

As mentioned C_e represents the equilibrium concentration of metal in solution and q_e (mg/g) is the content of metal adsorbed from solution in the equilibrium state. Q_o and b are Langmuir constants that depend on adsorption capacity and energy.

3. Results

Pilot tests represented that the removal efficiency of heavy metals is affected by pH parameter, contact time, metal concentration and adsorbent dose in water solutions. Also, the adsorption rate of each metal was investigated by adsorption isotherms which are represented below.

a. Adsorption isotherms

The Freundlich adsorption isotherm has been shown in Fig. 1. This figure shows that the logarithmic plot of q_e in terms of C_e is a straight line. Langmuir adsorption isotherms are represented below. The diagram of C_e/q_e in terms of C_e has been shown in Fig. 2.

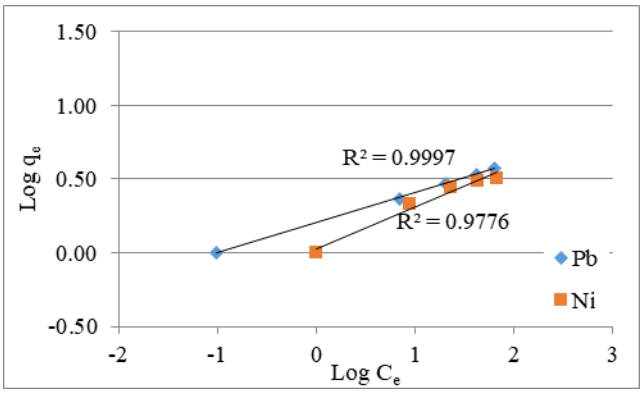

Fig. 1 Freundlich isotherms

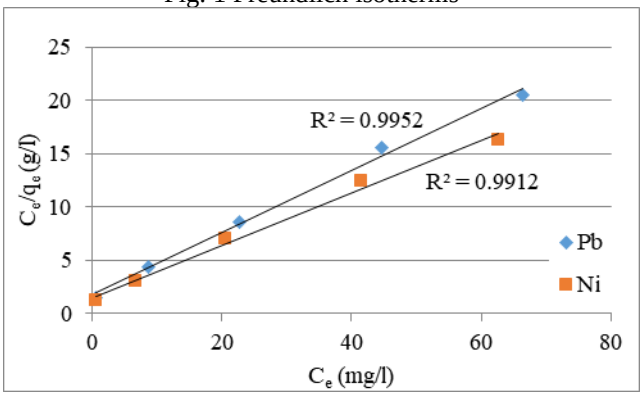

Fig. 2 Langmuir isotherms

Table 3 Correlation coefficients and constants of Langmuir and Freundlich isotherms

Metal	Freundlich isotherm			Langmuir isotherm		
	K_f	n	R^2	Q_0	b	R^2
Pb	1.60	4.95	0.99	3.80	0.30	0.99
Ni	0.98	3.22	0.97	3.46	0.20	0.99

According to Fig. 1, Fig. 2, and the correlation coefficients provided in Table 3, the adsorption rate of Pb R^2>0.99 and for the Freundlich and Langmuir isotherms and Ni R^2>0.97 and R^2>0.99 respectively for the Freundlich and Langmuir isotherms follow the adsorption isotherm equations. A method called Giles Classification is used in the qualitative description of adsorption isotherms (Giles et al. 1974). In this method, the q_e curve plots the content of metal adsorbed per unit weight of adsorbent in terms of the equilibrium concentration of metal in solution C_e.
Fig. 3 shows the adsorption isotherms of Pb and Ni ions.

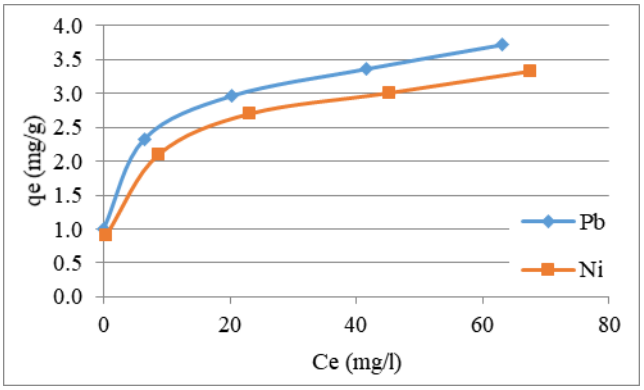

Fig. 3 Adsorption isotherms of Pb and Ni ions by activated alumina (pH=9, time=30min, adsorbent dose=0.5 g/l)

According to Fig. 3, by increasing the equilibrium concentration of metals in the solution, the adsorption rate of Pb and Ni ions per unit of adsorbent weight increases. There is also no significant difference between the adsorption of Pb and Ni ions. If the adsorbent tends to adsorb the desired ion, it is of type H, and if this tendency is moderate, it is of type L (Roman et al. 2020). According to Giles classification, the adsorption of Pb and Ni by activated alumina is of type H which means high interactions between metal ions and adsorbent.

b. Effect of initial concentration of metals

longer adsorbed. Fig. 4 shows that activated alumina efficiency of adsorbing Pb and Ni from aqueous solutions at a concentration of 25 mg/l for Pb and at a concentration of 20 mg/l for Ni is higher than other concentrations.

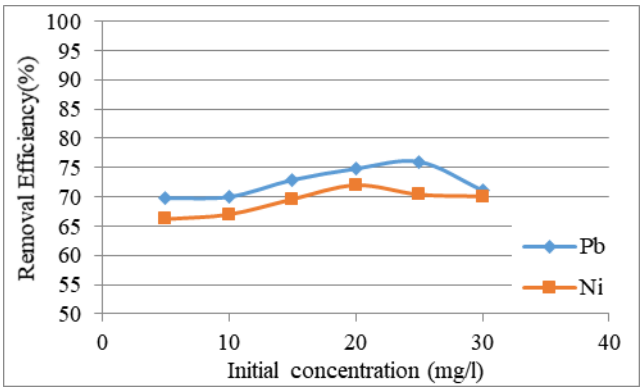

Fig. 4 Effect of content parameter of Pb and Ni concentration on the removal rate (pH=9, time=30 min, stirring speed=120rpm, size= 0.5mm, T=27^{0}C, adsorbent dose= 0.5 g/l)

c. Effect of initial pH

Effect of Variable pH parameter experiment was applied by pH range of 4-11 (acidic, neutral and alkaline pH range). To adjust the pH of the solution, sodium hydroxide (NaOH) and sulfuric acid (H_2So_4) were used. Fig. 5 shows the results obtained for the effect of pH on adsorption. The optimal pH is determined according to these results. The results indicated that the best adsorption occurs at a pH of 8 for Ni and 9 for Pb.

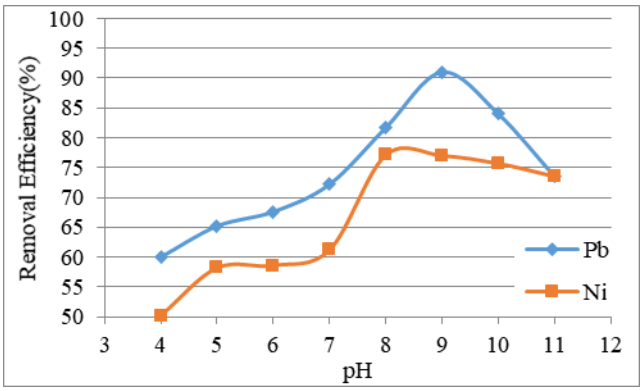

Fig. 5 Determination of the optimal pH of metal removal by alumina for Pb and Ni (C=20mg/l, time: 30min, Stirring speed=120rpm, size=0.5mm, T=27, adsorbent dose=0.5 g/l)

d. Effect of adsorbent dose

The next step is to determine the optimal content of activated alumina. Fig. 6 shows the results of the adsorbent dose parameter on adsorption rate. In the four studied content values of adsorbent (0.5, 2, 3 and 5 g/l), The highest adsorption occurred for Pb and then Ni for the adsorbent content of 3 g/l.

The effect of change in adsorption efficiency with initial concentration i

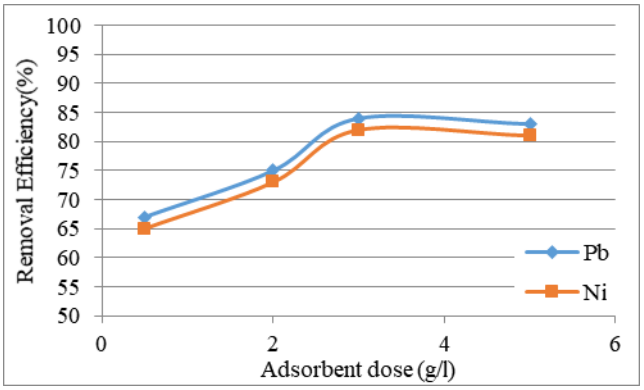

Fig. 6 Effect of the adsorbent content parameter on the removal rate (C=20 mg/l, pH=9, time=30min, stirring speed=120rpm, size=0.5mm, T=27^{0}C)

e. Effect of contact time

Determining the optimal contact time is investigated after determining the adsorbent content. Fig. 7 shows that in the studied times (15, 30, 60, 90 and 120 minutes) the best adsorption occurred at the contact time of 30 minutes, in other words, the equilibrium time of the removal process is approximately 30 minutes.

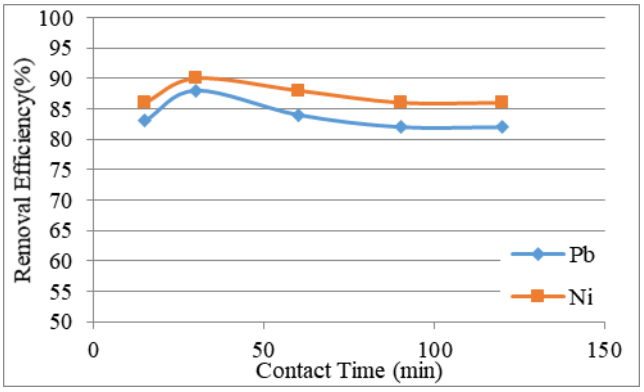

Fig. 7 Effect of contact time on the removal rate (C=20mg/l, pH=9, adsorbent dose=3 g/l, stirring speed=120rpm, size 0.5mm, T=27^{0}C)

4. Discussion

The findings of this study indicate that activated alumina adsorbs over 50 percent of dissolved Ni ions and over 60 percent of Pb ions in water solution. Also, the most effective parameter is pH which increased up to 8, Ni ions removal efficiency increased to 75 percent, pH increase up to 9, and Pb ions removal efficiency increases to 90 percent. Consequently, the optimum pH for water treatment would be the range of 8 to 9. In the following experiments pH as a constant value, has been considered 9. Heavy metal ions removal efficiency has been increased by change of other variables such as initial concentration, adsorbent dose and contact time over 65 percent. In other researches, activated alumina has been applied to remove heavy metal ions by filtration in a magnetic field (Szatyłowicz and Skoczko 2018). They investigated the use of a constant external magnetic field as an elementary additional process to improve the filtration efficiency on activated alumina for the removal of copper, lead and cadmium from water. Pilot tests showed that the use of activated alumina sorption materials with the magnetic field impact could decrease the copper, lead and cadmium content in the model water. Also, another study investigated that activated alumina could be efficient in the adsorption of textile dye (Wasti and Awan 2016). Batch adsorption experiments were carried out to see the effect of different parameters like initial concentration, contact time, stirring rate of the dye and dose of activated alumina. The removal efficiency of Cibacron reactive yellow dye with an initial concentration of 400 mg/L was greater than 90% for 90 min contact time. Freundlich and Langmuir adsorption isotherms were applied which fitted the data with an R^2 value of 0.99. Activated alumina also proved effective for the adsorption of dyes from actual textile wastewater giving a removal efficiency of 75%. Consequently activated alumina could be efficient to remove a different range of pollutants from water sources and wastewater which optimal conditions should be estimated by real field situation, pollution parameters and concentrations.

5. Conclusions

The results of this research, which is of experimental-research type, indicate that Langmuir and Freundlich isotherm models fitted well the adsorption data with the regression coefficient R^2 of 0.97 and 0.99 of lead and nickel. The research results corroborate that there is a significant difference between pH values of 8 and 9 and other pHs. Actually, at pH values above 9, nickel and lead ions react with hydroxyl ions due to the decrease of free H^+ ions in solution and the increase of free OH^- ions. Regarding the adsorbent dosage, it was found that activated alumina has a porous surface that can adsorb metals as surface adsorption, and the higher initial concentration of the metal solution and the adsorbent content in the solution leads to a higher adsorption percentage. Also, the equilibrium time of the removal process was determined to be about 30 minutes. The optimal points of the adsorption process in optimal conditions for lead are pH of 9, concentration of 25 mg/l, adsorbent dose equal to 3 g/l. Results represent that optimal conditions for Nickel removal are pH of 8, concentration of 20 mg/l, adsorbent dose of 3 g/l and contact time of 30 minutes. Finally, activated alumina can be used as an effective and inexpensive adsorbent to remove lead and nickel heavy metals from water sources.

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7. Conflict of interest

The authors declare that they have no conflict of interest.

8. Additional Information and Declarations

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Grant Disclosures

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Competing Interests

The authors declare there is no competing interests, regarding the publication of this manuscript.

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