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Porous Silicon Powder as an Adsorbent of Heavy Metal (Nickel)

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Abstract. New and inexpensive nanoporous silicon (NPS) powder was prepared by alkali chemical etching using sonication technique and was subsequently investigated as an adsorbent in batch systems for the adsorption Ni(II) ions in an aqueous solution. The optimum conditions for the Ni(II) ion adsorption capacity of the NPS powder were studied in detail by varying parameters such as the initial Ni(II) concentration, the solution pH value, the adsorption temperature and contact time. The results indicated that the maximum adsorption capacity and the maximum removal percent of Ni(II) reached 2665.33 mg/g and 82.6%, respectively, at an initial Ni(II) concentration of 100 mg/L, adsorption time of 30 min and no effect of the solution pH and adsorption temperature.

INTRODUCTION

Water contamination caused by heavy-metal ions generated from alloys, pigments, electroplating, mining, metallurgical activities, nuclear power plant operations, aerospace industries, electrical contacts, printing, and the manufacture of paper, rubber, plastics, and batteries is a global problem receiving worldwide attention. The extended persistence of water contamination in biological systems and the tendency to bioaccumulate while moving up the food chain is a serious threat to human health, living resources, and ecological systems. [1]

The heavy metal toxicity caused by industrial waste water and other natural sources has become a threat to the environment and ecosystem for the past many decades. Very small concentration of heavy metal present in water increases the health problems to humans and animals. Nickel (Ni) is one of the non biodegradable, toxic, heavy metal present in waste water and ground water. There are various conventional treatment methods for removal of heavy metals from water and waste water. [2] The conventional methods that are employed to remove heavy metals such as ion exchange, chemical precipitation, reverse osmosis and membrane separation are found to be inefficient and expensive, especially when treating wastewater with low concentration of heavy metals. Adsorption, on the other hand, has emerged as a potential alternative to conventional physicochemical technologies in waste-treatment facilities. Adsorption is an effective separation process that has advantages in terms of cost, flexibility and simplicity of design, and ease of operation compared to other techniques. Adsorption also does not result in the formation of harmful substances. [3] The adsorption process offers flexibility in design and operation of treatment processes as well as producing high-quality treated effluent in many cases. Adsorbents which have large surface area, pore volume and proper functionalities can be expected to perform more effectively. [4]

Ni is the 24th most abundant element in the Earth's crust and is used in many industrial and commercial applications including electroplating, battery manufacture, forging, metal finishing and mining, all of which lead to environmental pollution by Ni. [4] It's one of the important toxic metals; Ni(II) finds its way into the water bodies through effluents from industries. The Ni salts are known to be acutely and chronically toxic to human. Acute poisoning of Ni(II) causes headache, dizziness, nausea, and tightness of the chest, chest pain, shortness of breath,

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dry cough, cyanosis, and extreme weakness. At higher concentrations it is a potent carcinogen and causes cancer of the lungs, nose and bone. [3]

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Nanoporous silicon (NPS) is a versatile material whose fundamental properties are of value to diverse fields, tunable surface chemistry, huge surface area, and controllable morphology. For this reason and due to its variable pore-size, NPS is a suitable host material for loading other nanosized structures, e.g., small molecules, metals, and magnetic nanoparticles, thereby adding properties of the confined spaces to the pores. [5] For this reason, NPS is considered one of the best elements used in the process of removing heavy materials such as nickel due to the raw materials mentioned in previous studies. [6, 7] The present study includes the adsorption studies on Ni(II) using powder of NPS. The efficiency of this adsorbent was studied and maximum adsorption and lowest equilibrium time for this adsorbent was recorded.

MATERIALS & METHODS

NPS Powder Preparation as an Adsorbent Material

The sonication technique is used in this work, because of its simplicity. To prepare (NPS) powder, we used a commercially available Si-powder [Silicium, Pulver – 99%]. It was prepared via wet alkali chemical etching using the sonication technique. Briefly, appropriate amount of Si powder was dispersed in a solution of KOH, n-propanol and distl. H2O. The resulting powders were filtrated and washed, and then dried overnight at 40°C to obtain NPS powder. The structure of producing powder is characterized by XRD (X-ray 7000 Schimadzu diffractometer). XRD equipped with a Cu anode operated at 40 kV and 30 mA; the samples were scanned at a rate of 4 °/min.

Preparation of Metal Ion Solutions

The Ni(II) was synthesized using standard methods. The double distilled water was used for all the analyses. The concentrations of the metal ions were estimated using UV-Visible Spectrophotometer Double Auto cell (Labomend. INC, USA). Standard Nickel Solution: Ni(II) solution was prepared by using AR Grade nickel chloride; 0.2 g of Nickel chloride was taken into a 1000 ml volumetric flask.

Batch Procedure for Ni Element Removal

Batch Equilibrium Method: All experiments were carried out at room temperature (27 °C) in batch mode. Batch mode was selected because of its simplicity and reliability. The experiments were carried out by taking 40 ml metal ion sample (AR grade) in a 100 ml Erlenmeyer flask and after pH adjustments; a known quantity of solution dried adsorbent (NPS) was added. The flasks were agitated at 200 rpm for predetermined time intervals using a mechanical shaker until equilibrium conditions were reached. After shaking, the suspension was allowed to settle. The residual biomass adsorbed with metal ions, the filtrate was collected and subjected for metal ion estimation using UV- Visible Spectrophotometer Double Auto cell (Labomend. INC, USA). The values of percent metal uptake by the sorbent (Sorption efficiency) and the amount of metal ion adsorbed has been calculated using the following relationships: [8]

$$Sorption\ efficiency = \frac{C_i - C_f}{C_i} \times 100 \qquad \text{Eq.1}$$

$$Amount\ Adsorbed\ (Q_e) = \frac{C_i - C_f}{W} \times V \qquad \text{Eq.2}$$

Where, Ci= Initial concentration of metal ion in the solution (mg/L), Cf = Final concentration of metal ion in the solution (mg/L), W = Weight of adsorbent (g), V = Volume of solution (L) and Qe =Amount of metal ion adsorbed per gram of adsorbent.

RESULTS & DISCUSSION

Adsorbent Powder Study

The XRD pattern of fabricated NPS powder, before using it as an adsorbent, is shown in FIGURE 1. All the diffraction peaks can be well indexed to the cubic phase NPS reported in (JCPDS Card No. 01-079-0613 and 00-027- 1402). Before removal process, the strongest peak was appeared at 2θ = 28.23 ° correspond to (111), and other peaks were appearing at 2θ = 47.193 °, 56.023 ° and 69.13 °, which correspond to (220), (211) and (400), respectively. No diffraction peaks arising from any impurity can be detected in the pattern confirming that high-purity NPS powdered material has been obtained [6], {the crystalline size (1- 3 nm), the void size (0.2- 0.3 nm) and porosity (10- 30 %)}. Either after the Ni-removing process using NPS powder, noticeable, the appearance of the diffraction peaks at 2θ = 76.68 °, 88.34 °and 95.24 °, arising from the Ni (220), (201) and Ni (311) planes (JCPDS Card No. 45-1027), respectively.

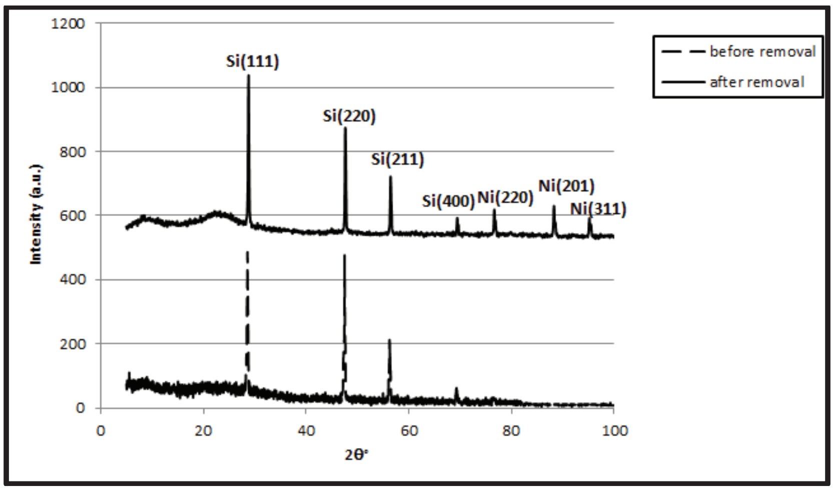

FIGURE 1. X-ray diffraction patterns of (a) NPS powder; (b) adsorbed Ni on the NPS powder

Adsorption Study

Uptake of the Ni^{2+} by NPS as a function of contact time in different Ni concentrations is illustrated in FIGURE 2. As it can be seen, in all of the Ni concentrations (30, 50, 100, 200 ppm), removal process took place. Ni was removed fast; it exhibited a subsequent removal until equilibrium was reached. The main reason for the appearance of rapid process, that as a result of the plenty active sites on the NPS powder, the adsorption process and gradual occupancy of these sites causes of emerging [9]. In fact Ni ions, form a molecule (actually ion) layer on the adsorbent surface. After that, the uptake rate is controlled by the rate at which the Ni^{2+} ions are transported from the exterior to the interior sites of the NPS powder particles. [10]

Effect of initial Ni(II) concentration on adsorption:

The initial Ni(II) concentration serves as an important driving force for overcoming mass transfer resistance of Ni(II) between the aqueous and solid phases. The effects of different initial Ni(II) concentrations on the NPS powder adsorption capacity are shown in FIGURE 2. The adsorption capacity of the NPS powder toward Ni(II) first increased and then decreased with increasing initial Ni(II) concentration over 100 ppm at contact time 30 min, noticeable, the removing percent of Ni(II) at several concentration is nearly similar values. Studies were carried out to obtain optimum conditions for the adsorption of Ni(II) using 0.03 g of adsorbent (NPS) in a 1L solution of 100 ppm of Ni(II) concentration adjusted to different contact time values from 5 to 30 min, which produce Ni removal percent is 82.6% and the value of the adsorption capacity of NPS is 2665.3 mg/g. This result was observed because higher Ni(II) concentrations result in an increased concentration gradient, which, leads to a higher probability of collision among Ni(II) ions and the active adsorption sites on the NPS powder, thereby increasing adsorption capacity. With further increases in Ni(II) concentration than 100 ppm, the adsorption capacity remained constant because the active adsorption sites became saturated. [11]

Effect of contact time on adsorption:

The effects of different adsorption times on the NPS powder adsorption capacity toward Ni(II) are shown in FIGURE 3. At prolonged adsorption times, the Ni(II) adsorption capacity of the NPS powder initially increased rapidly and then decreased slowly because the surface of the NPS powder was covered with a large quantity unsaturated functional groups. Ni(II) ions were adsorbed by diffusing into the microporous adsorbent (NPS); thus resulting in a sharp adsorption equilibrium that decreased with the saturation of the functional groups on the NPS powder surface. [12] Therefore, the optimum contact (adsorption) time is 30 min, the studies were carried out to obtain optimum conditions for the adsorption of Ni(II) using 0.09 g of adsorbent (NPS) in 1L solution of 30 mg/L of Ni(II) concentration adjusted to different contact time values from 5 to 30 min, which produce Ni removal percent is 98.4 % and the value of adsorption capacity of NPS is 862.9 mg/g.

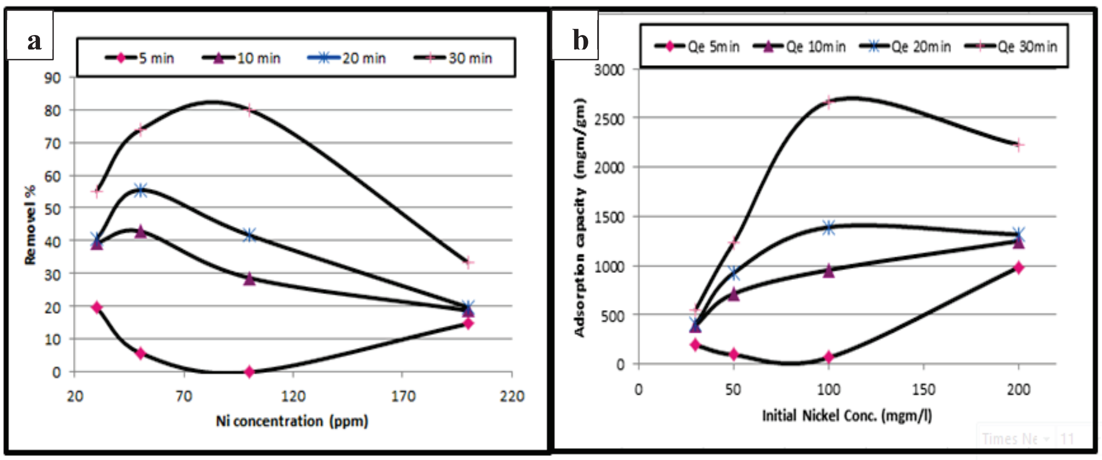

FIGURE 2. At the NPS powder dose 0.03 g/L, a) Ni removal concentration percent as a function of Ni concentration (ppm) b) Effect of Ni concentration on desorption capacity of NPS.

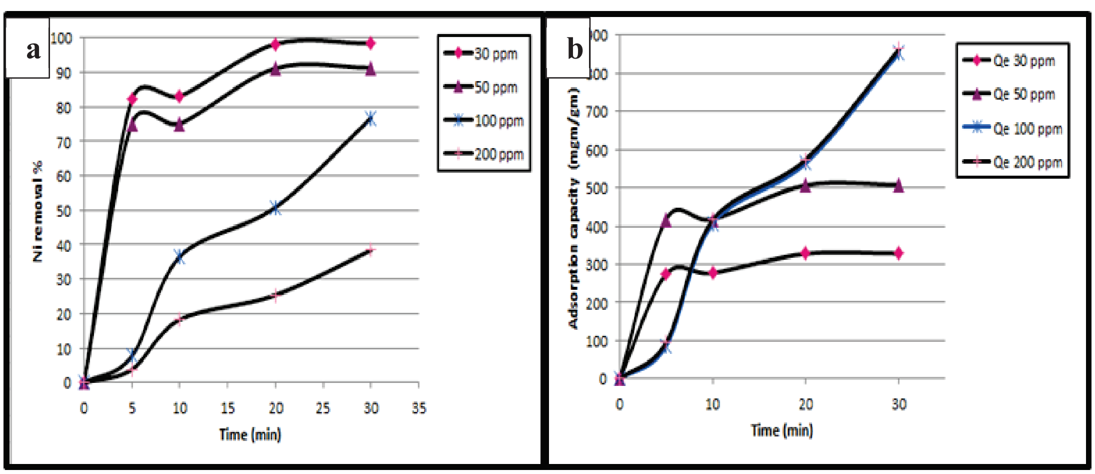

FIGURE 3. At the NPS powder dose 0.09 g/L, a) Ni removal concentration percent as a function of contact time (min), b) Effect of contact time on desorption capacity of NPS.

In addition, the effect of acidity and temperature on the adsorption value of nickel was studied. It has been shown that by changing the pH value and temperature values, there is no obvious effect at the varying process of the Ni adsorption values, which are the same as in case of the room temperature and pH = 7.

CONCLUSION

A new NPS powder was prepared by alkali chemical etching using the sonication technique. The results of this study showed that the synthesized NPS powder could be used effectively for the adsorption of Ni(II) ions from aqueous solutions. The maximum Ni(II) ion adsorption capacity for the NPS powder reached to 2665.3 mg/g under an initial Ni(II) concentration 100 mg/L and adsorption time is 30min, in addition, no effect of pH and at adsorption temperature. Noticeable, the minimum value of contact time is 5 min, it is enough to removing 82.12 % of initial concentration of Ni(II) 30 mg/L.

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