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Original Research Paper

Heavy metal ions adsorption and photodegradation of remazol black XP by iron oxide/silica monoliths: Kinetic and equilibrium modelling

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ABSTRACT

The adsorption of heavy metal ions (Cr^{3+} , Pb^{2+} and Cd^{2+}) by metal oxide monoliths (Fe_2O_3 and $\text{Fe}_2\text{O}_3/\text{SiO}_2$) synthesized via nanocasting method using SiO_2 monoliths as a template was studied. The adsorption experiments were performed in different batches by varying key parameters and the equilibrium between the adsorbents and metal ion solution was achieved in ~ 120 min at pH 6. The maximum monolayer adsorption efficiency for Pb (II), Cr (III) and Cd (II) ions was 850, 770 and 690 mg/g, respectively, for the magnetic $\text{Fe}_2\text{O}_3/\text{SiO}_2$ monoliths. The experimental data show best fit with the pseudo-second-order reaction type. The adsorption data found to be well fitted using Freundlich and Langmuir adsorption isotherms. The adsorption process was exothermic and spontaneous in nature, as confirmed by the thermodynamic parameters. Furthermore, the photocatalytic degradation of an industrial dye e.g., remazol black XP (RXP) by $\text{Fe}_2\text{O}_3/\text{SiO}_2$ monoliths was done from wastewater and the photocatalytic efficiency of the monoliths (using different amount) has been evaluated under visible light source which gives the best results (97.8%) for the monolith concentration 0.10 g/L.

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1. Introduction

Heavy metal ions and dye stuffs in wastewater were considered as the most serious environmental problems. To reduce the environmental toxicant load, it is required to remove the heavy metal ions, organic dyes, etc. from the wastewater because of their non-biodegradable, toxic nature and serious effectiveness on the aquatic as well as soil ecological systems. The water pollutants cause risks for human health as well as hinder aquatic life and, as a result, their potential health risks induce many problems like disorder in the cell cycle, multiple organ damages and may lead to carcinogenic diseases [1–4]. The significant amount of heavy metals like chromium, mercury, lead, cadmium etc. are obtained from the wastewater from leather, textile, pigment & dyes, tannery, paint, wood processing, photographic film production and petroleum refining industries [5]. Apart from this, surface treatment and electroplating practices lead to the creation of huge amount of wastewater containing heavy metals like lead, chromium, cadmium, nickel, silver, vanadium, platinum, copper and titanium [6].

Chromium is largely found in industrial wastewater and it is also applied in different industrial work like metallurgy, chrome

tanning, dyes, leather industries and electroplating operations [7,8]. Along with, chromium also found in groundwater while leaching soil in mines [9]. Generally, chromium stable in two major oxidation states i.e. Cr (III) and Cr (VI), whereas Cr (VI) compounds are more harmful (10- to 1000- fold) and more soluble than Cr (III) compounds [10]. Also, different sources like electroplating, photographic materials and leather industries [11,12] produce Cr (III) which readily diffuses in the environment and accumulates in living cells [13,14]. Accidental chromium ingestion causes stomach upsets, ulcers, kidneys and liver damages, and even death. The WHO (World Health Organization) has set a limit for maximum 0.05 mg/L in drinking water for chromium due to its toxicity and carcinogenicity [15]. So, the heavy metal ions removal from wastewater and groundwater is very necessary to avoid the contamination in the water sources and make it suitable for public supply. Up to date, various techniques have been developed like reverse osmosis and nanofiltration, coagulation/coprecipitation, ion exchange, cryogenic separation, adsorption, and membrane separation [16–18] for the heavy metal ions removal. Among them, adsorption is very popular as commonly used technique due to its simplicity and cost-effectiveness [19]. However, selection of the adsorbent makes it successful technique. The biosorbents used in the adsorption process develop a huge amount of secondary waste and unable to discharge standard alone. Also, they are difficult to

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separate out from the solution for the separation process and hence insufficient effective. Therefore, there is need to develop new adsorbents which can be easily synthesized and exhibit good adsorption capacity and stability with extraordinary regenerative ability.

Several metal oxides including MgO, CuO, Fe₂O₃, ZnO, TiO₂, MnO₂ etc. have been investigated for the heavy metal ions adsorption [20,21]. These metal oxide adsorbents are easy to prepare, safe and inexpensive to produce as well as they are attractive due to their surface active properties. The iron oxide-based porous adsorbents also show the promising results for heavy metal ion removal (e.g. lead, chromium, zinc etc.) from wastewater with remarkable catalytic properties [22,23]. On the other hand, the magnetic nature of these adsorbents is very useful for the separation process because, on applying the magnetic field, the adsorbent can easily be separated from the solution [22]. The combination of this advantageous magnetic property of iron oxide materials with the meso/micro-porous silica exhibits high adsorption capacity. The preparation of iron oxide materials surrounded by porous silica adsorbents has been reported for the heavy metal ions removal by several research groups [24–27]. Heidari et al. found the adsorption capacities of 57.74, 18.25 and 12.36 mg/g for Pb (II), Cd (II) and Ni (II) by modified MCM-41 [28]. Egodawatte et al. [22] synthesized magnetic iron oxide/silica nanocomposites for Cr (III) adsorption and found 36.92 mg/g of adsorption capacity at pH of 5.4 whereas, on further modifying by amino-propyl, they obtained highest adsorption capacity of 108.16 mg/g.

In the literature, it can be seen that mostly synthesized metal-oxide adsorbents are in powder form and that has some drawback like it generates a miscible suspension with water which demands high-quality filters and instruments for its extraction from the solution. Also, it was found that there is not so much research done on the synthesis of the metal oxides using nanocasting method which is very useful for controlling the textural properties of the adsorbents. So, in this regard, the objective of the present study is to synthesize magnetic Fe₂O₃ and Fe₂O₃/SiO₂ monoliths (single rock-like structure) using nanocasting technique for the heavy metal ions adsorption and photodegradation of the industrial dye, RXP. The effects of pH, temperature, contact time and adsorbent concentration for the heavy metal ion removal were also explored. Furthermore, adsorption kinetics, isotherm and thermodynamic studies were also performed in details. Likewise, an effort has been made to study the photocatalytic efficiency and reduction rate for the RXP dye under visible light using a different weight of Fe₂O₃/SiO₂ monoliths.

2. Experimental section

2.1. Materials

Octadecyltrimethylammonium bromide (CTAB, 99%) and polyethylene glycol (PEG, >97%) were obtained from Sigma-Aldrich, US. Ferric nitrate (98%), ammonia (28–30%) and nitric acid (69%), were obtained from Merck, India. Chromium (III) nitrate (97%), lead nitrate (99%), and cadmium carbonate (98%) were obtained from Spectrochem, India. Here, synthetic wastewater was prepared by mixing of metal ions (lead, chromium, and cadmium) and organic dye, RXP. Milli-Q water (18.2 MΩ cm) was used for the preparation of all the synthetic solutions. The organic dye, RXP was collected from the local textile industry, Ludhiana, India.

2.2. Synthesis of SiO₂ monoliths

The preparation of the silica monolith was done by sol-gel method [29]. Initially, nitric acid (30%) was added to the solution

of PEG (9.54 × 10⁻⁴ M) and water (8.25 ml). TEOS (8.1 ml) was added after the dissolution and the solution was stirred for 10–15 min at room temperature. Then, CTAB surfactant was dissolved into the solution under the continuous stirring. The achieved sol was shifted to microplates and kept for 72 h at 40 °C to reach sol to gel transformation. The resultant monoliths were soaked in NH₄OH solution (1M) at 90 °C for 9 h and then washed with 0.1 M nitric acid solution for acidification. Later, monoliths were washed with de-ionized water by 3–4 times and kept for drying in oven at 40 °C for 4 days. The monoliths were then calcined at 550 °C for 5 h at a rate of 1 °C/min.

2.3. Synthesis of Fe₂O₃/SiO₂ monoliths

Initially, the SiO₂ monoliths were degassed for 2 h and then the aqueous solution of 50 ml, 2 M Fe (NO₃)₃ was impregnated. Then, the monoliths were heated for 9 h at 120 °C. These two steps were repeated for 3 times for the proper impregnation of the solution into the silica monoliths. Finally, the monoliths were calcined at 600 °C for 2 h with a heating rate of 5 °C/min.

2.4. Synthesis of Fe₂O₃ monoliths

To attain the pure Fe₂O₃ monoliths, the above-obtained monoliths was treated with 100 ml of 2 M NaOH solution for 24 h, and then washed repeatedly with water by 2–3 times followed by drying at 70 °C for 24 h. The synthesis flow diagram for the monoliths is shown in Fig. 1.

2.5. Characterization methods

The pore sizes and BET surface area of the adsorbents were obtained from BET surface area analyzer (Bel, Japan, Inc, Microtec BELSORP MINI-II). The PSD (pore size distribution) curves were calculated by BJH method (Barrett-Joyner-Halenda). X-ray diffractometer (PANALYTICAL X'Pert PRO) was used for the XRD (X-ray diffraction) analysis with Cu Kα (λ = 1.540 Å) radiation operated at 45 KV. Morphology and size of the monoliths were studied by SEM (scanning electron microscope), JEOL JSM-6510 LV at 20 KV accelerating voltage. The XPS analysis (X-ray photoelectron microscopy) spectra were recorded with monochromatized aluminium source (ka) source of 1486.7 eV energy. Magnetic measurements of the monoliths were carried out by vibrating sample magnetometer (VSM) Lake Shore (VSM) 7404. The analysis of the metal ions concentrations was performed using Atomic adsorption spectrophotometer (AAS) of GBC 932AA. The kinetic parameters of dye degradation were monitored with a UV-Vis spectrophotometer (Champion UV-500).

2.6. Adsorption study

The adsorption experiments were conducted at room temperature under favorable conditions. All of the three monolithic adsorbents (SiO₂, Fe₂O₃/SiO₂ and Fe₂O₃), each 0.02 g in weight, were inserted into the 100 ml of various synthetic metal ion solutions (Cr (III), Cd (II) and Pb (II)) with initial concentration of 10 mg/L. The respective solutions with the different adsorbents were kept at various time periods with the speed of 200 rpm and then centrifuged (5000 rpm) for 10 min. The residual metal ion concentration of the supernatant solutions was detected by AAS. The adsorption capacity (q_e) of the monoliths (mg/g) was calculated by using Eq. (1).

$$q_e = \frac{(C_o - C_e)}{m} \times V \quad (1)$$

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