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Synthesis, Characterization of ZnO and Al_2O_3 Nanoparticles and Its Application in Chromium Remediation Studies

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Present work describes the synthesis of zinc oxide and aluminum oxide nanoparticles by simple chemical method. The synthesized nanoparticles were characterized using XRD, SEM and TEM. Both ZnO and Al_2O_3 nanoparticles were used in the remediation of chromium from aqueous solution. Various parameters such as pH, volume (mL)/mass (g) (v/m) ratio, contact time and initial concentration of chromium were evaluated to optimize the removal efficiency of chromium. The obtained results present that both the nanoparticles acted as potential adsorbents in removing chromium from aqueous solution, maximum removal was observed at pH 5. The contact time required to achieve the equilibrium was 60 min for ZnO and 95 min for Al_2O_3. Under optimum process parameters, the removal efficacy was maximum and reusability of the synthesized nanoparticles was also checked.

Keywords: ZnO nanoparticles, Al_2O_3 nanoparticles, Cr(VI) solution, Remediation studies.

INTRODUCTION

Metal and metal oxide nanoparticles (MONPs) represent an era of change and has given a new momentum in studies of material sciences towards research and health-related applications. From past decade, metal oxide nanoparticles have gained a lot of attention due to their applications in gas sensors, piezoelectric devices, field emission devices, power generators, ultraviolet light emitters, sensors, coatings and paints [1-4]. Different synthesis methods of these MONPs have been reported in the literature such as chemical vapor deposition [5], thermal decomposition [6], sol-gel methods [7] and microemulsion method [8]. But these methods are expensive when compared to aqueous medium based methods which are also simple and cost effective. Rapid industrialization has led to heavy metals contamination of water sources such as mercury, lead, cadmium and chromium, which originate from mining activities, petroleum refining, paint industries and leather factory's, *etc*. [9]. Chromium is the main industrial metal used in various processes and released into the environment through poor storage and improper disposal practices. Chromium in the environment occurs in two different oxidation states among which hexavalent chromium [Cr(VI)] is toxic and carcinogenic [10]. Adsorption technology has been increasingly used in the removal of toxic metals from wastewaters. Many natural compounds such as algae, rubber, wood, *etc*. have been used as adsorbents. However new economical and highly effective adsorbents are still needed. The present results stated that the synthesized nanoparticles of zinc oxide and aluminum oxide acted as potential adsorbents in the removal Cr(VI) from aqueous solution.

EXPERIMENTAL

Synthesis of ZnO and Al_2O_3 nanoparticles: 100 mL of 0.01 N zinc acetate dihydrate solution was taken in a beaker kept in an ice bath and 100 mL of 0.2 N NaOH was added dropwise with stirring at 600 rpm for 2 h. The resulting turbid solution was heated on a water bath at 75 ºC for 30 min, then settled white powder was separated followed by washing with deionized water thrice and dried overnight at room temperature.

Similar procedure was followed to synthesize Al_2O_3 nanoparticles wherein 1.72 g of aluminum nitrate was dissolved in 470 mL of water, then 30 mL liq. NH_3 was added drop by drop with vigorous stirring at 550 rpm. Finally obtained powder was calcined at 700 ºC for 2 h.

Characterization of ZnO nanoparticles: The synthesized nanoparticles were initially checked by UV-visible spectroscopy (JASCO-V-670, Shimadzu) (Fig. 1a-b). Figure 1a shows a peak at 362 nm for ZnO anoparticles and Fig. 1b shows a peak at 270 nm for Al_2O_3 anoparticles. Before removal study the stability of ZnO and Al_2O_3 nanoparticles was checked by determining zeta potentials of their aqueous dispersion using Horiba Scientific Nano Particii (SZ-100). Fig. 2 represents the zeta potential analysis results of ZnO nanoparticles (Fig. 2a)

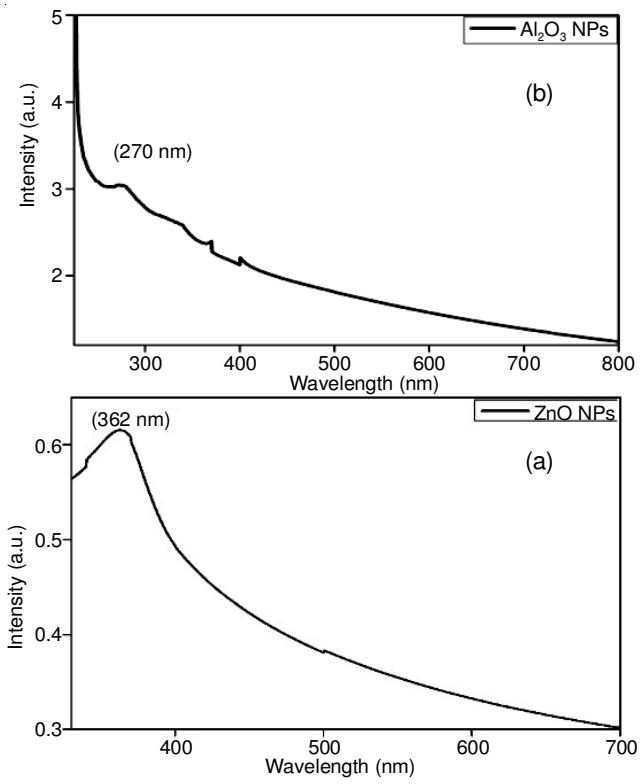

Fig. 1. UV-visible spectra of ZnO NPs (a) and Al_2O_3 NPs (b)

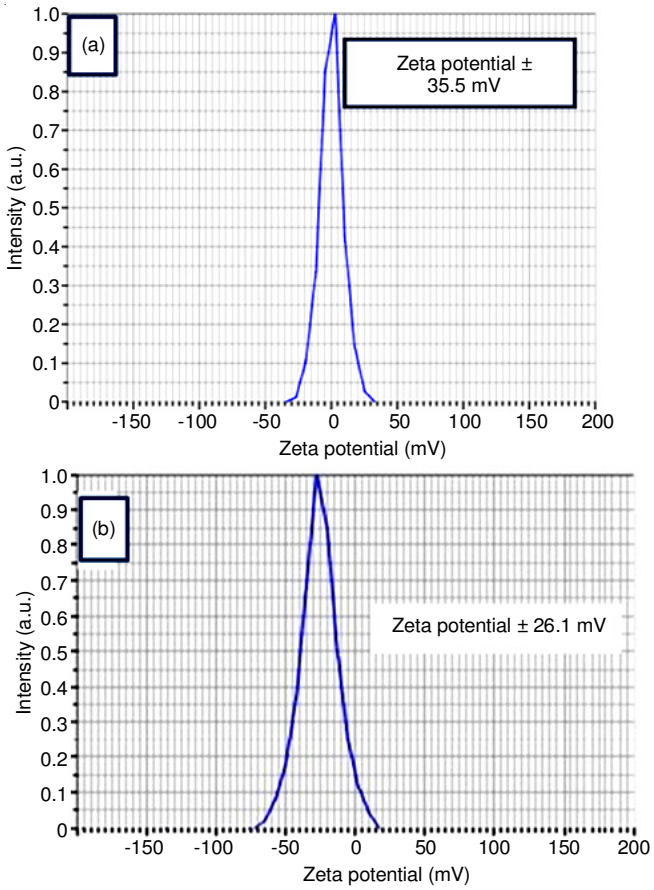

Fig. 2. Zeta potential analysis of ZnO NPs (a), Al_2O_3 NPs (b)

and Al_2O_3 nanoparticles (Fig. 2b). Zeta potential of ZnO nanoparticles dispersion was -26.1 mV and that of Al_2O_3 nanoparticles was ± 35.5 mV, which suggests high stability of these nanoparticles.The synthesized solid materials were analyzed by Powder X-ray Diffraction (XRD) (Bruker D8 Advance Diffractometer, Bruker AXS, Germany) with Cu Kα radiation (k = 1.54 Å). Scanning Electron Microscope (SEM) analysis was done using a Carl Zeiss SEM instrument (model No: EVOMA 15, Oxford instrument).

Effect of different Optimization parameters on removal of Cr(VI) from aqueous solution

Effect of pH on removal efficiency: In order to study the effect of pH, 100 mg/L Cr(VI) solution was prepared and from which 20 mL of solution is taken into four different reaction tubes and the pH is adjusted to 3, 4, 5 and 6, respectively. 100 mg of ZnO nanoparticles were added to each bottle and kept for spinning at 50 rpm for 4 h in a roto-spin. After spinning, the solution is filtered and then the concentration of chromium is estimated by AAS. Another set of the reaction was carried out with and Al_2O_3 nanoparticles with a similar procedure.

Effect of v/m ratio on removal efficiency: 20 mL of Cr(VI) solution (100 mg/L) was taken into four different reaction tubes and the pH is adjusted to 5. The varying amount of ZnO nanoparticles were added *i.e.*, 50, 100, 150 and 200 mg. Then the bottles were Roto-spinning at 50 rpm for 4 h, then solutions were filtered and AAS analysis was carried out. Similarly, another set of the reaction was carried out with varying amount of Al_2O_3 nanoparticles.

Effect of contact time in removal of Cr(VI) solution: 20 mL of Cr(VI) solution (100 mg/L) was taken into five different reaction tubes and the pH is adjusted to 5 in each one of them. Then 150 mg of ZnO nanoparticles were added to each bottle. The bottles were kept for spinning at 50 rpm for 2, 4, 8, 18 and 24 h, then solutions were filtered and AAS analysis was carried out. In case of Al_2O_3 nanoparticles, 200 mg were added in all the tubes.

Effect of initial concentration on removal efficiency: 100, 200, 400 and 600 mg/L of Cr(VI) solutions were prepared and from which 20 mL of each solution is taken into four different reagent bottles and maintaining th pH 5. 150 mg of ZnO nanoparticles were added to each bottle. Then these bottles were kept for spinning at 50 rpm for 4 h and finally the solutions were filtered and subjected to AAS. A similar reaction with 200 mg of Al_2O_3 nanoparticles was carried out.

RESULTS AND DISCUSSION

Fig. 3A shows the XRD pattern of the synthesized ZnO nanoparticles and all the peaks in the XRD pattern were matched with hexagonal ZnO nanoparticles (JCPDS Card No. 89-0510). The XRD pattern clearly showed the hexagonal phase structure, and strong diffraction peaks revealed that the synthesized ZnO nanoparticles were highly crystalline. The SEM images of synthesized ZnO nanoparticles at different magnifications are shown in Fig. 3B and 3C. It is very clear that well dispersed ZnO nanoparticles were formed with the average particle size of 152.34 nm.The XRD pattern of Al_2O_3 nanoparticles (Fig. 4A) showed peaks corresponding to α-Al_2O_3 (JCPDS card no: 83-2080). The strong diffraction peaks indicated the formation of crystalline nanoparticles. Fig. 4B and 4C shows the SEM images of Al_2O_3 nanoparticles at different magnifications which confirm the formation of needle shaped Al_2O_3 nanoparticles.

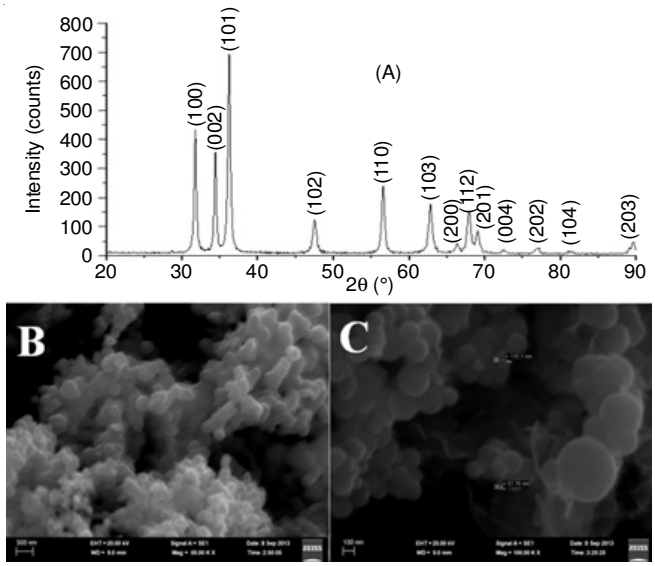

Fig. 3. XRD pattern of synthesized ZnO NPs (A) SEM images of ZnO NPs at 300 nm scale(B) 100 nm scale (C)

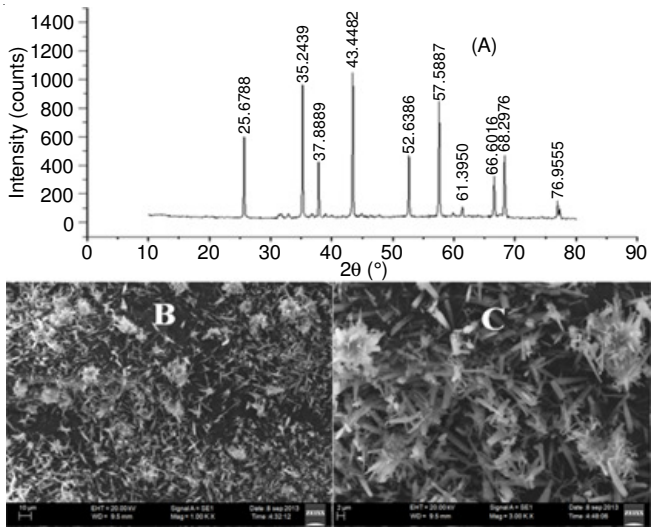

Fig. 4. XRD pattern of synthesized Al_2O_3 NPs (A) SEM images of Al_2O_3 NPs at 10 μm scale (B) and 2 μm scale (C)

The adsorption study was carried out and different process

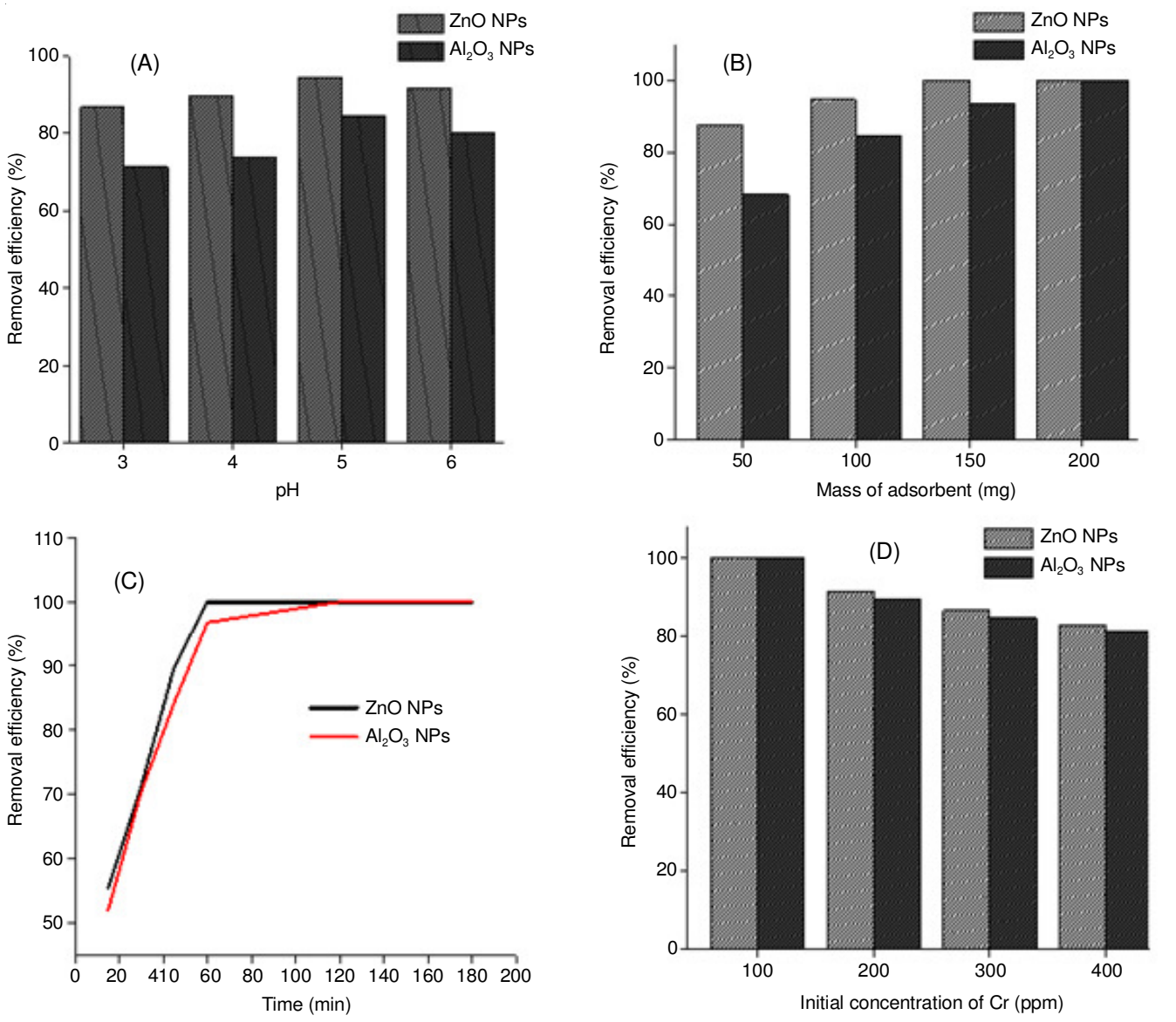

Fig. 5. Graphs showing the removal efficiency of Cr using ZnO and Al_2O_3 NPs with different parameters pH (A), v/m ratio (B), contact time (C), initial conc. of Cr(VI) (D)

parameters were optimized (Table-1 and Fig. 5). The maximum removal efficiency was found at pH 5 (Fig. 5A), Hence, the pH 5 was maintained throughout the experiment. As the v/m ratio increased the removal efficiency decreased (Fig. 5B). The time required to reach equilibrium was found to be 60 min and 90 min for ZnO and Al_2O_3 nanoparticles, respectively (Fig. 5C). By addingdifferent amount of synthesized nanoparticles to Cr(VI)solution, it was observed that maximum removal efficiency was achieved at 150 mg for ZnO nanoparticles and 200 mg for Al_2O_3 nanoparticles (Fig. 5D). As the initial concentration of Cr(VI) increased the removal efficiency decreased in both the cases (Fig. 5D).

TABLE-1
DIFFERENT OPTIMIZED PARAMETERS FOR REMOVAL OF Cr(VI) BY ZnO AND Al_2O_3 NPs

Different optimized parameters	ZnO NPs	Al_2O_3 NPs
pH	5	5
v/m ratio	133	100
Time (min)	60	90
Concentration (ppm)	100	100

Conclusion

Synthesis of ZnO and Al_2O_3 nanoparticles was achieved by simple chemical method without using any stabilizing agents. XRD results showed that the synthesized ZnO nanoparticles were highly crystalline and formation of α-Al_2O_3 nanoparticles. Further synthesized ZnO and Al_2O_3 nanoparticles were used as an adsorbent in chromium remediation studies; maximum removal was found to happen at pH 5. For the studies on v/m ratio, highest removal efficiency was optimized at 150 mg of ZnO nanoparticles and 200 mg of Al_2O_3 nanoparticles in 20 mL chromium solution keeping the constant volume at 50 mL for each.

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