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Green synthesis of magnesium oxide nanoflower and its application for the removal of divalent metallic species from synthetic wastewater

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Abstract

Present manuscript deals with the green synthesis of magnesium oxide nanoflower by using bio-compatible acacia gum. MgO nanoparticles were synthesized by the chemical precipitation method and then treated with two different concentrations (10% and 20%) of acacia gum (AG) to develop flower shaped MgO nanoparticles. Magnesium oxide nanoflower was characterized by various techniques such as XRD, TEM, SEM and atomic force microscopy (AFM). XRD confirms the formation of crystalline MgO. TEM analysis demonstrated that particle size of acacia gum treated MgO nanoparticles was higher in comparison to untreated *n*-MGO particles which confirm coating of acacia gum on bare *n*-MgO. AFM also supports the formation of nanosized particles. Synthesized samples were then used as adsorbent material for the abatement of divalent metallic ions viz. Co(II), Cd(II), Zn(II), Cu(II), Mn(II), Pb(II) and Ni(II) from synthetic wastewater. Magnesium oxide nanoflower was found to be very efficient in the removal of all selected divalent metallic species.

Keywords: Adsorption; Acacia gum; Magnesium oxide; Nanoflower; Precipitation

1. Introduction

During the last decade, oxide based nanomaterials have received considerable attention due to their unique physical and chemical properties [1–5]. Among various metal oxides, magnesium oxide (MgO) is the most promising candidate due to its unique and excellent optical, electrical, thermal, mechanical and chemical properties and also its high ionic character [6]. At nanoscale, MgO shows high reactivity because of the presence of large number of highly reactive edges, structural defects on the surface, unusual lattice planes and high surface to volume ratio [7]. MgO has a very high melting point and low heat capacity, which makes it suitable candidate for insulation applications. MgO has numerous applications in various fields such as catalyst supports, agricultural products, paints, superconductor products, antimicrobial materials, photonic devices, refractory materials, electro-optical devices, sensors and as adsorbent [6–11]. Recently, the application of MgO nanoparticles as adsorbent materials has attracted more scientific interest due to their high removal efficiency [12]. MgO nanoparticles act as destructive adsorbent for chemical species and this property increases with decrease in MgO size [7]. Various methods have been developed for the synthesis of nanosized MgO such as sol–gel method, laser ablation, thermal decomposition, chemical precipitation, spray pyrolysis, sonochemical synthesis, hydrothermal, thermal evaporation, solvothermal reaction, microwave-assisted synthesis method, flame metal combustion, electrochemical, combustion, chemical vapour deposition etc. [7,10,11,13,14].Various studies are presently available for the surface modification of MgO nanoparticles for various applications. But in most cases, the use of toxic surfactant, coating material may further increase the possibility of nanotoxicity. To overcome this problem, a biocompatible, nontoxic and readily available biopolymer, acacia gum, has been used for development of flower like structure of *n*-MgO. Acacia gum is the dried exudate obtained from certain species of acacia tree and shows high solubility, good rheological properties, emulsion stability, pH stability, non-toxicity

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and gelling characteristics [15–17]. The main component of acacia gum is polysaccharide, protein and arabino-galacto protein species with charged groups. Due to presence of charged groups, acacia gum shows tendency of intramolecular hydrogen bonding and shows anionic character. It is reported that acacia gum enhances the adsorption efficiency of any adsorbent [17,18].

In this paper, we have described a simple method to synthesize MgO nanoflower via chemical precipitation. MgO nanoparticles were treated with acacia gum for the development of flower like structure. The resulting products were characterized by X-ray diffractometer (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM), particle size analyzer, thermo gravimetric analysis (TGA). pH_{zpc} of nano-flowers were also determined for better illustration of surface complexation with different metallic species. Synthesized nanoflower was used for the removal of divalent metallic species viz. Co(II), Cd(II), Zn(II), Cu(II), Mn(II), Pb(II) and Ni(II) from synthetic wastewater.

2. Material and method

2.1. Synthesis of magnesium oxide nanoflower

For the synthesis of MgO nano-flower, 0.2 M Mg^{2+} solution was prepared by adding $MgCl_2.6H_2O$ salt in distilled water. Thereafter, 0.5 M NaOH solution was added drop by drop into Mg^{2+} solution. The reaction mixture was continuously stirred (600 rpm) on a magnetic stirrer at room temperature till complete process of precipitation. After precipitation, the precipitate was separated from the solution by centrifugation at 5000 rpm for 15 min. The precipitate was washed with distilled water and dried in hot air oven at 60 °C for 48 h. Dried $Mg(OH)_2$ precipitate was grounded in tube mill and calcined in a muffle furnace (NABERTHERM B-170) at 500 °C for 3 h by keeping heating rate 5^0 C/min to obtain magnesium oxide nanoparticles (*n*-MgO). Calcined *n*-MgO particles were treated with acacia gum for its surface modification. Two different concentrations of acacia gum viz. 10% and 20% were prepared for MgO modification. *n*-MgO (5 g) was added into 100 mL of 10% and 20% solution of acacia gum separately. Both samples were sonicated for 30 min in sonicator which was followed by 4 h shaking at 25^0 C in temperature controlled shaker at 200 rpm. After shaking, nanoparticles were separated by centrifugation and washed with water and dried in hot air oven (TERMAKS) at 50 °C for 24 h. Dried powders were grounded in tube mill (IKA Tube mill control) to remove lumps. *n*-MgO treated with 10% and 20% solution of acacia gum was named as AGM-10 and AGM-20, respectively. $Mg(OH)_2$ precipitate was characterized by XRD and TGA to confirm the formation of $Mg(OH)_2$ particles. *n*-MgO, AGM-10 and AGM-20 samples were characterized by XRD, TEM, SEM, AFM and particle size analyzer. pH_{zpc} of AGM-10 and AGM-20 nanoflower was also determined by reported method [19]. For determination of pH_{zpc} of AGM-10 and AGM-20, 0.20 g of both samples were added separately into NaCl solutions of different pH ranging from 2.0 to 12.0 and kept for 48 h. After 48 h, adsorbent was separated from NaCl solution and final pH of NaCl solution was measured by pH meter (INOLAB WTW Series). The point of intersection of 'pH_{final} vs $pH_{initial}$' curves was recorded as pH_{zpc} of nanoparticles.

2.2. Adsorption experiment

Synthesized AGM-10 and AGM-20 nanoflowers were used as adsorbent material for the treatment of divalent metallic ions viz. Co(II), Cd(II), Zn(II), Cu(II), Mn(II), Pb(II) and Ni(II) from synthetic wastewater. Synthetic wastewater (200 mg/L) was prepared by adding appropriate salt of selected metals into distilled water. For investigation of removal efficiency of AGM-10 and AGM-20 nanoflowers, 2 g/L of both samples were added into metal rich synthetic wastewater and agitated at room temperature in a temperature controlled shaker for 1 h by maintaining agitation speed at 180 rpm. To observe the effect of modification on removal efficiency of *n*-MgO, batch experiments were also carried out with *n*-MgO. After 1 h shaking, adsorbent was separated from the solution by centrifugation (Eppendorf 5810R) and residual concentration of ions was measured by Inductively coupled plasma optical emission spectrometer (ICP-OES), Model: iCAP 6300 (Thermo Electron Corporation). Experiments were carried out in

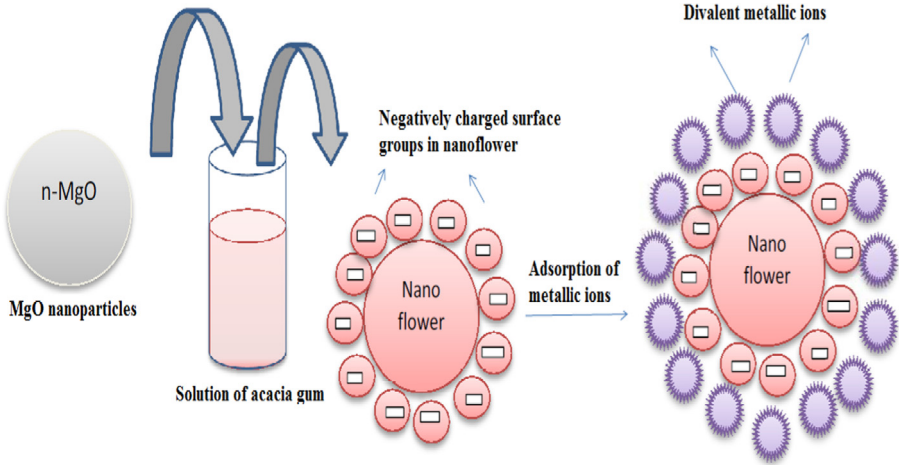

Fig. 1. Mechanism of nanoflower synthesis and adsorption.