ABSTRACT

Water quality has become a major global concern due to ever increasing human development activities, depletion of natural resources and pollution of surface and groundwater. The drastic environmental changes and rapid industrialisation led to change of water quality. Water consumption also grew rapidly due to expanding industrial production, increasing population and intensive agricultural production. Hence there is a growing need to tackle water pollution problem. During the past few years, nanoparticles have been gaining much attention for adsorption of pollutants due to their unique physico-chemical properties. Magnetic nanoparticles are especially found to be handy because of easy separation. An inert support is required to enhance the efficiency of the nanoparticles. Among the various supports examined for adsorption studies graphene appeared to be a suitable support.

In the present investigation, magnetic Fe$_3$O$_4$ core-shell nanorods (MCSNs), reduced graphene oxide supported nanocomposites (RGO-NC) and reduced graphene oxide supported Fe$_2$O$_3$/TiO$_2$ nanocomposite (RGO-MFT) were synthesized and characterized for their physico-chemical properties. These materials were attempted for the removal of As(III), As(V) and Hg(II) from aqueous solution. The adsorption experiments were carried out by batch studies. The parameters like pH, adsorbent dose and contact time were
optimised for the maximum removal of arsenic in the batch studies. Adsorption isotherms and kinetic studies were also carried out to evaluate the nature of diffusion process.

Magnetic Fe$_3$O$_4$ core-shell nanorods (MCSNs) were synthesized by crosslinking amine functionalized Fe$_3$O$_4$@SiO$_2$ core-shell nanoparticles with 1,2-bromochloroethane. These nanorods were then protonated with dilute HCl. The MCSNs nanorods were characterized by FT-IR, N$_2$ adsorption, VSM, SEM and TEM. The surface area of MCSNs nanorods (335 m$^2$/g) was higher than bare Fe$_3$O$_4$ and amine functionalized Fe$_3$O$_4$@SiO$_2$. These nanorods were used simultaneously as ion-exchanger and adsorbent for the removal of arsenic from aqueous solution. It exhibited high adsorption capacity for arsenic. The kinetic study revealed that adsorption equilibrium attained within five min. The adsorbed arsenic on the nanorods were removed by magnetic separation and regenerated by acid treatment. The percentage removal of arsenic was found to be more than 99%.

Graphene oxide was synthesized by Hummers method and then reduced with hydrazine hydrate to obtain reduced graphene oxide. Dihalogen crosslinked Fe$_3$O$_4$ core-shell nanoparticles (DCSNs) were synthesized by crosslinking amine functionalized Fe$_3$O$_4$@SiO$_2$ core-shell nanoparticles with 1,2-bromochloroethane. These nanoparticles were then loaded on reduced graphene oxide to obtain reduced graphene oxide- nanocomposites
(RGO-NC). The nanocomposites were characterized using FT-IR, Raman, N\textsubscript{2} sorption, VSM, SEM and TEM. The surface area was found to be 388 m\textsuperscript{2}/g due to crosslinking of amine functionalized Fe\textsubscript{3}O\textsubscript{4}@SiO\textsubscript{2} core-shell nanoparticles with 1,2-bromochloroethane. The saturation magnetization of RGO-NC (17.1 emu/g) was sufficient to remove the nanocomposites from aqueous solution. The nanocomposites were used as adsorbent for the removal of arsenic and mercury from aqueous solution. The maximum adsorption capacity for As(V) at pH 6 was found to be 45.5 mg/g whereas at neutral pH the adsorption capacity for As(III) and Hg(II) was 62.7 and 81.3 mg/g respectively. The adsorption capacity of arsenic and mercury was slightly affected by the coexistence of anions and cations respectively.

Mesoporous Fe\textsubscript{2}O\textsubscript{3}/TiO\textsubscript{2} was synthesized by sol-gel route using triblock copolymer (pluronic P123) as the structure directing agent. The mesoporous nature of the material was confirmed from low angle X-ray diffraction. The material was supported on reduced graphene oxide and characterized by FT-IR, Raman, XPS, N\textsubscript{2} sorption, HR-TEM and SEM. The reduced graphene oxide supported Fe\textsubscript{2}O\textsubscript{3}/TiO\textsubscript{2} nanocomposite was tested for the sorption of both As(V) and As(III) from aqueous solution. The equilibrium adsorption attained within fifteen minutes for both As(III) and As(V). The maximum adsorption capacity for As(V) and As(III) was found to be 99.5 and 77.7 mg/g at pH 6 and 7 respectively. The sorption was found to be high in the pH range of 6-7. The low adsorption at lower and higher pHs other than 6 and 7 was due to zero point
charge of the nanocomposite. The recyclability study of nanocomposite confirmed the stability and reusability.

The amine functionalized nanorods (MCSNs) and reduced graphene oxide supported amine functionalized nanoparticles (RGO-NC) can also be used for the adsorption of carbon dioxide and other hazardous pollutants. The slight modification of reduced graphene oxide supported mesoporous metal oxide nanoparticles (RGO-MFT) can also be used as visible light active photocatalyst for water decontamination.