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Metal oxides as adsorbent for lead ion

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Oxide materials specially metal oxide nanomaterials exhibits good adsorbent property which develops materials science and technology at nano level. The better adsorbent property for the oxide nanomaterials is due to the particle size. The decrease in particle size of the metal oxide particles develops new properties and applications due to variation of surface morphology. Copper oxide and zinc oxide was synthesized by microwave firing through the thermal decomposition of copper carboxylate and nickel carboxylate precursors employing polyvinyl alcohol as an efficient fuel. The prepared oxide materials are studied for its adsorbent behavior for lead ions by dynamic method. Lead adsorbed oxide materials is characterized for its structure by employing powder X-ray diffraction (XRD) pattern, morphology by Scanning Electron Micrograph (SEM) tool and bonding by Fourier Transform infrared (FTIR) tools. Solution conductivity and Atomic absorption study for lead eluent is studied.

Keyword: Nanomaterials, Adsorption, Bonding, Structure, Morphology

1. Introduction

Fine particles of metal oxide materials increases the surface area and also increases the adsorption sites. Porous oxide materials prepared employing different synthetic routes[1-3], shows excellent adsorption behavior for heavy metal ions. In case of aluminium oxide the octahedral aluminum sites are fully occupied while vacant sites are randomly disturbed at the tetrahedral positions. These vacant sites in the structure are mainly responsible for the adsorption behavior on γ-Al_2O_3. Similar case can also be observed in iron oxide sample[4]. In many environmental settings, the presence of organic ligands that form stable complexes with metal cations can strongly influence their adsorption behavior on metal oxides, including those of Al and Fe. For example, Co(II) forms strong complexes with ligands such as ethylenediaminetetraacetic acid (EDTA) and nitrilotriacetic acid (NTA), and complexes of this type sorb to oxide surfaces in a fashion analogous to that of bare ligands (e.g. EDTA, NTA) rather than the metal cation[5-6]. The adsorption of these complexes increases with decreasing pH, until a pH is reached where the proton-promoted dissolution of the solid releases sufficient structural metal to induce competitive complex dissociation. Fe and Al oxide surfaces show high affinity for natural organic molecules containing carboxylate functional groups[7]. These surfaces may be coated with a veneer of sorbed organic molecules in soils and aquatic sediments. Adsorption of Co (II) on both γ-Al-$(OH)_3$ (gibbsite) and α-FeO[OH] (goethite), as well as on natural materials containing these oxides, is enhanced by the presence of complexed organic matter which appears to function as a co-complexant along with the oxide surface[8].

Present work reports the adsorption behavior of copper oxide and nickel oxide prepared by microwave route. The dynamic method is used for adsorption study of lead ion on metal oxides. The lead adsorbed metal oxide samples are well characterized for its structure by X-ray diffraction (XRD), morphology by Scanning Electron Microscope (SEM) and bonding by Fourier Transform Infrared study (FT-IR) techniques. Solution conductivity and Atomic absorption study for lead eluent is studied.

2. Experimental

2.1 Materials and method

Lead acetate chemical used in the present study is of AR grade. The dynamic method is adopted for the adsorption study. Metal oxides are prepared by microwave route using polyvinyl alcohol as an efficient fuel.

2.2 Adsorption Study

The preparation of copper oxide and nickel oxide sample by microwave route using polyvinyl alcohol as a fuel is reported in our earlier work [9]. One gram of prepared copper oxide and nickel oxide samples were taken into the 100 ml conical flask and 50 ml of known concentration of lead acetate solution was added in to the conical flask. Pack the conical flask mouth with aluminium paper followed by cotton. Shake the content of the conical flask solution in mechanical shaker for 12 hours for adsorption of lead on metal oxides. After complete adsorption, the lead solution was eluted and preserved for atomic absorption studies. The solid product i.e., adsorbent air-dried and is characterized employing XRD, IR, and SEM studies and eluent lead solution is studied for solution conductivity and Atomic absorption study.

2.3 Characterization

The structures of as prepared nickel oxide were studied by X – ray diffraction using Phillips X – ray diffractometer (PW3710) with Cu Kα as source of radiation. Morphology and bonding of the above oxide was studied by Phillips XL 30 ESEM and Perkin–Elmer 1600 spectrophotometer in KBr medium tools respectively.

3. Results and discussion

3.1 X-ray diffraction studies

Figure 1(a-b) shows the XRD pattern of lead adsorbed copper oxide and nickel oxide sample respectively. This pattern shows Bragg's reflections corresponding to lead ions, which is adsorbed on the surface of oxide particles. Both patterns shows the presence of respective metal oxide peak along with lead peaks. The lead peaks in the pattern are identified by JCPDS file no 44-0872. Presence of lead peaks with oxide peaks confirms adsorption behavior of lead on metal oxides.

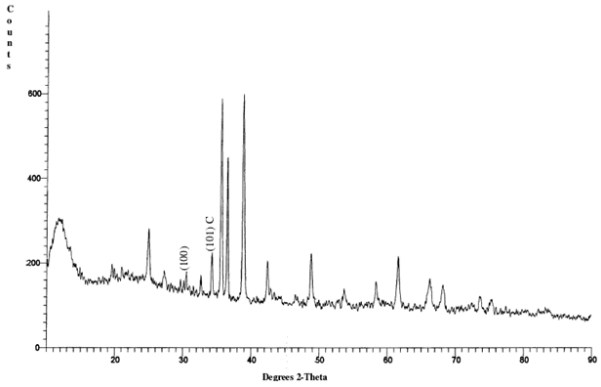

Fig 1(a): XRD Pattern of Lead adsorbed CuO

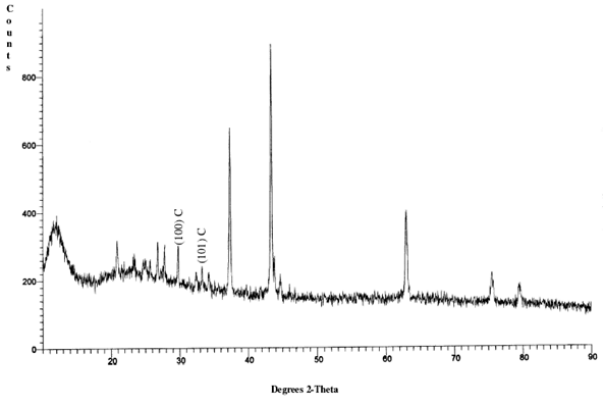

Fig 1(b): XRD Pattern of Lead adsorbed NiO

3.2 Scanning Electron Micrograph

Figure 2(a-b) shows SEM image of lead adsorbed copper oxide sample at low and high resolution respectively. The image shows fie and irregular particle morphology with globular arrangement. The close netting with formation of loops can observed on the high resolution image. Figure 3(a-b) shows SEM image of lead adsorbed nickel oxide sample at low and high resolution respectively. Self-assembled spherical lead particles are also observed on the surface oxide particles. Fine continuous lead particle packing can also be observed. However on higher resolution the same can be observed clearly. This tool clearly shows the lead adsorption on the surface of metal oxide materials and is supported by earlier XRD results.

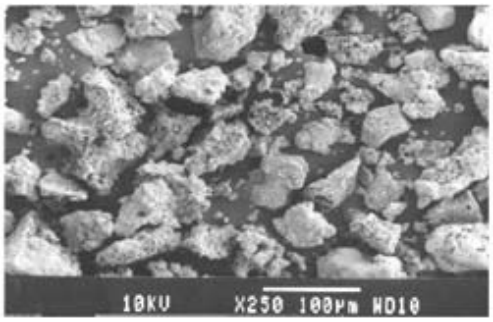

Fig 2(a): SEM image of Lead adsorbed CuO sample at low resolution

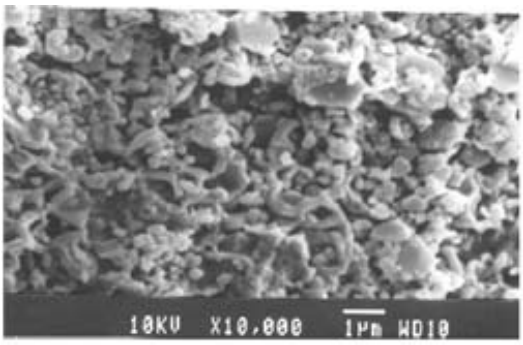

Fig 2(b): SEM image of Lead adsorbed CuO sample at high resolution

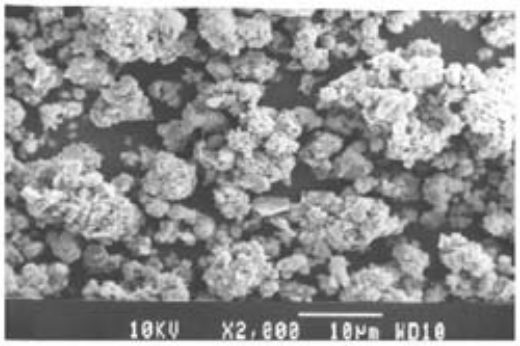

Fig 3(a): SEM image of Lead adsorbed NiO sample at low resolution

3.3. Infrared studies

Table 1 shows the vibrational frequencies of lead adsorbed on copper oxide and nickel oxide samples. Both samples shows the vibrational frequency at 3000 cm^{-1} corresponds to water of hydration and the IR bands at around 1600 is characteristic peak of the acetate group.

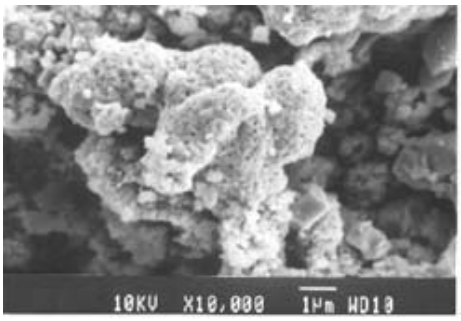

Fig 3(b): SEM image of Lead adsorbed NiO sample at high resolution

Bands at 1080 is assigned to in plane bending of CH_3 group. Both adsorbed oxide sample shows Frequency below 100cm-1are due to the presence of metal-Oxygen vibrational frequency[10]. Careful observation of IR spectra of pure metal oxides and its lead adsorbed samples shows the new bands are weak in nature in the latter case, which indicates a weak interaction of the adsorbed Pb^{2+} ions on the oxide samples.

Table 1: Vibrational Frequencies of Lead adsorbed CuO and NiO

Samples	Lead adsorbed CuO	Lead adsorbed NiO
Frequencies	3600,1080,550,510	3200,1620,1080,480

3.4 Atomic Absorption Study and Conductivity Results

The results obtained by atomic absorption study and solution conductivity study are given in table-2. An atomic absorption spectroscopic characterization was carried out for the blank lead acetate solution and the eluent solution after adsorption of CuO and NiO samples. The initial concentration of lead acetate solution is 280ppm, whereas after adsorption, the concentration of lead solution decreases to 107.5ppm for copper oxide and 108.2ppm for nickel oxide sample respectively. This decrease in concentration of eluent solution indicates the absence of some lead ions in the eluent lead solution and confirms the adsorption of lead ions on the metal oxides.

Solution conductance gives information about the conducting nature of pure lead acetate as well as eluent lead solutions of copper oxide and nickel oxide samples. The concentration goes on decreasing, the conductance increases because of rapid movement of the metal ions. The conductance of pure lead acetate solution is 3.2X 10-5mho-1, eluent solution of CuO is $5.1X10^{-5}$ and that of NiO is 5.3mho-1 respectively.

This increase of conductance indicates the absence of some lead ions in the eluent solution and confirms the adsorption of lead ions on the zinc oxide.

Table 2: AAS and Solution Conductivity results of Lead adsorbed CuO and NiO samples

Parameters	Concentration of Lead acetate solution	Concentration of lead after passing through CuO	Concentration of lead after passing through NiO
AAS (ppm)	280	107.5	108.2
Solution Conductivity (mho^{-1})	$3.2X\ 10^{-5}$	$5.1X\ 10^{-5}$	$5.3X\ 10^{-5}$

4. Conclusions

The nanosized metal oxide shows better adsorbent for heavy metal ions like lead. This dynamic method shows good method for adsorption study is may be applicable for other heavy metal ions like mercury ion. Hence, this method can also adopt for the adsorption of heavy metal ions on other nanosized metal oxide.

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6. Reference

1. Towle SN, Bargar JR, Brown JGE, Parks GA. Surface Precipitation in the Aqueous Co(II)/Al_2O_3 System. J Colloid Int Sci 1997; 187:62.
2. Bargar JR, Towle SN, Brown JGE, Parks GA. Structure, Composition, and Reactivity of Pb(II) and Co(II) Sorption Products and Surface Functional Groups on Single-crystal a-Al_2O_3J. Colloid Int Sci 1997; 185:473.
3. Bargar JR, Brown JGE, Parks GA. Surface Complexation of Pb(II) at Oxide-Water Interfaces: I. XAFS and Bond-valence Determination of Mononuclear and Polynuclear Pb(II) Sorption Products on Aluminum Oxides Geochimica. Acta 1997; 61:2617.
4. Lagashetty A, Vijayanand H, Basavaraja S, Mallikarjuna NN, Venkataraman A. Lead adsorption study on combustion derived g-Fe2O3 surface. Bull Mater Sci 2010; 33(1).
5. Brown GE. Metal Oxide Surfaces and Their Interactions with Aqueous Solutions and Microbial Organisms. Chem Rev 1999; 99:77.
6. Arunkumar L, Mahesh B, Venkataraman A. Adsorption study of Pb^{+2} ions on Nanoceramics. J Matl & Mater Sci 2010; 52(4):357-362.
7. Mallikarjuna N, Venkataraman A. Adsorption of Pb2+ ions on nanosized γ-Fe2O3: formation of surface ternary complexes on ligand complexation. Talanta 2003; 60:139.
8. Zachara JM, Smith SC, Kuzel LS. Adsorption and dissociation of Co-EDTA complexes in iron oxide-containing subsurface sands. Geochim Cosmochim Acta 1995; 59:4825.
9. Lagashetty A, Havanoor V, Basavaraj S, Balaji SD, Venkataraman A. Microwave-assisted route for synthesis of nanosized metal oxides. Sci & Tech Adv Mater 2008; 8:484-493.
10. Rao CNR. Chemical applications of infrared spectoscopy, Academic Press, New York and London, 1963.