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SYNTHESIS, CHARACTERIZATION AND SORPTION BEHAVIOR OF NOVEL ION EXCHANGE MATERIALS FOR THE TREATMENT OF INDUSTRIAL WASTE WATERS

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Abstract: *In this paper we describe the analytical application for the treatment of industrial waste waters by ion exchange materials. The structural studies reveal semi-crystalline nature of the material with the particle size up to 20µm. Physiochemical properties such as ion-exchange capacity, chemical and thermal stability of composite material have also been determined. Bifunctional behavior of the material has been indicated by its pH titrations curves. The material exhibits improved thermal stability, higher ion-exchange capacity and better selectivity for important metals ions. The ion-exchange material shows an ion-exchange capacity of 2.52 meq g-1 for Na^+ ions. Sorption behavior of metal ions on EDTA Ti(IV)phosphate was studied in DMW, different concentrations of solvents such as acetic acid or methyl cyanide and surfactant systems. The cation exchanger was found to be selective for Pb^{2+}, Fe^{3+}, Th^{4+}, Cd^{2+}, Mg^{2+} and Ni^{2+} ions. The limit of detection was found to be 9.12 (0.0020), 1.78 (0.0010), 0.88 (0.0013) and 1.05 (0.0004) µgL−1 for Ni^{2+}, Cd^{2+}, Cu^{2+} and Fe^{3+} ions. Analytically necessary separations of metal ions in synthetic mixtures moreover as industrial effluents and natural water were achieved with the exchanger. The material has been synthesized via sol-gel and was characterized on the basis of FTIR, TGA/DTA, XRD and SEM studies.*

Key Words: *EDTA, Sorption, FTIR, TGA/DTA, XRD and SEM.*

1. INTRODUCTION:

Analytical chemistry is taken into account because the Mother of Science it plays an important role in every field namely Biology, Physics and Biotechnology. It prospers because it adapts to changes in chemical science and focuses on improvements in experimental design, chemo metrics and the creation of new measurement tools to provide better chemical information.

Modern analytical chemistry covered main aspect as identification, quantification, elucidation of structure, separation of different elements in natural and artificial materials. It also focused on improvements in experimental design, chemo metrics and the creation of new measurement tools to provide better chemical information. Separation process has very important applications in various fields such as medicine, agriculture and environmental analysis. Drinking Water Inspectorate reported that tap water may contain contaminants, including toxic metals as well as pesticides, drugs and chemical waste from industry [1].

In recently, contamination of water with heavy metal ions has become a major area of concern. These are frequently introducing to the atmosphere through the industrial discharges [2-5] and feebly affecting the lives of living beings. Additionally, considerable quantities of heavy metals released into the environment through routes other than in aqueous form in wastewaters such as lead, nickel, copper, zinc, chromium, cadmium and mercury are widely used in electroplating, batteries, water pipes, alloys.

Annually, huge amount of heavy metals are produced from the mining of their respective ores. In 21st century, approximately $14{,}500\times10^3$ tons of copper was produced [6]. Plants need these metal ions in micronutrient level higher concentrations are known to produce toxic effects. High exposure levels of lead causes encephalopathy, cognitive impairment, behavioral disturbances, kidney damage, anemia and toxicity to the reproductive system [7]. Cadmium is associated with nephrotoxic effects particularly at high exposure levels; long term exposure may cause bone damage [8]. High concentrations of mercury lead to neurobehavioral disorders, developmental disabilities including dyslexia, attention deficit hyperactivity disorder, and intellectual retardation [9]. Excessive copper concentrations can leads to weakness, lethargy and anorexia as well as damage to the gastrointestinal tract [10]. With regard to heavy metal pollution, there have been growing interests in the development of novel organic-inorganic ion-exchange materials which are competent to remove metal ions even in low concentration from the contaminated water. In our laboratory a number of titanium based hybrid ion exchangers have been synthesized [11-13] that possessed all such characteristics discussed earlier

and are highly selective for heavy metals ions in the environment samples however EDTA Ti(IV)phosphate cation exchanger possess excellent ion exchange capacity along with thermal and chemical stability compared to the reported titanium based ion exchangers.

2. RESULTS AND DISCUSSIONS:

We tend to describe the acceptable condition for the synthesis of composite ion-exchanger. Number of samples of poly-o-toluidine Zr (IV) tungstate were prepared by sol-gel method. The ion-exchange capability of the synthesized material depends upon the pH and the mixing ratio of the reactants. It is inferred from with increasing pH 0.7 to 1.5.

3. EXPERIMENTAL SECTION:

Materials and methods

Zirconium (IV) oxychloride, sodium tungstate, ortho-toluidine (Merck limited). The other reagents used were of analytical grade. 0.1M solutions of zirconium (IV) oxychloride (A) and sodium tungstate (B) were prepared in DMW while 20% solutions (v/v) of ortho-toluidine and potassium persulphate (0.1M) were prepared in 1.0 M HCl.

Apparatus

A digital pH meter Elico EL-10 (Elico India) was used for pH measurements. Shimadzu Graphicord UV-240 spectrophotometer with 10-mm matched quartz cells for spectrophotometric determination, Infrared (IR) spectra was recorded on a Fourier Transform-IR Spectrometer from Perkin Elmer (1730, USA) using KBr disc method. Thermo gravimetric Analysis/Differential Thermal Analysis (TGA/DTA) analysis was carried out by DTG –60 H; (Schimadzu,) analyzer at a rate of 10ºC min^{-1} in nitrogen atmosphere. An X' Pert PRO analytical diffractometer, Holland with radiation λ= 1.5418 Å) was used for X-ray diffraction measurement. Scanning Electron Microscope (SEM) instrument was used for SEM images of the material at different magnifications. Transmission Electron Microscopy analysis was carried out by Jeol H-7500 Microscope. FAAS measurements were made with a Model GBC-932-Plus flame atomic absorption spectrometer. A temperature-controlled shaker was used for shaking. Muffle furnace was used for heating samples at different temperatures.

Selective separation of Ba^{2+} and Hg^{2+} ions from the synthetic mixtures (Fe^{3+}, Cu^{2+}, Al^{3+}, Sr^{2+}, Ca^{2+}, Zn^{2+}, Ba^{2+}) and (Ca^{2+}, Sr^{2+}, Fe^{3+}, Cu^{2+}, Cd^{2+}, Hg^{2+}) was achieved on poly-o-toluidineZr(IV)tungstate columns. The amount of the Ba^{2+} and Hg^{2+}ions in the mixture was varied by keeping the amount of the other metal ions constant. (Shown in Table-1)

Table 1: Effect of temperature on the ion-exchange capacity of EDTA Ti (IV) phosphate cation exchanger on heating for 1 h.

Temperature (ºC)	Color	%Weight loss	% Retention of IEC
25	White	0.0	100
50	White	0.0	100
100	White	0.0	100
200	Brown	2.0	98
300	Black	10	92
400	Black	36	71
500	Light brown	44	62

4. CONCLUSION:

Recently synthesized semi crystalline EDTA Ti (IV) phosphate cation exchanger shows sorption behavior towards heavy metal ions and can withstand fairly high temperature with. 98% retention of ion uptake capacity up to 200°C. It has been used for the quantitative separation of binary mixture of metal ions of analytical importance. Determination of Ni^{2+}, Cd^{2+}, Cu^{2+} and Fe^{3+} ions from natural and industrial waters by FAAS does not need any prior

digestion. The material can be explored further for the removal and recovery of metal ions from industrial waste waters. The EDTA Ti (IV) phosphate exchanger, thus, exhibits the characteristics of a promising ion-exchanger which might be explored for the treatment of waste waters.

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