

**AMBERLITE XAD-2 BASED CHELATING RESINS :
SYNTHESIS AND THEIR BEHAVIOUR AS
METAL ION EXTRACTANTS**

by
REENA SAXENA (nee SAHAY)

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**DEDICATED
TO MY
HUSBAND**

CERTIFICATE

This is to certify that the thesis entitled, "**AMBERLITE XAD-2 BASED CHELATING RESINS: SYNTHESIS AND THEIR BEHAVIOUR AS METAL ION EXTRACTANTS**" being submitted by **Mrs. Reena Saxena**, to the Indian Institute of Technology, Delhi, for the award of the degree of Doctor of Philosophy in Chemistry is a record of bonafide research work carried out by her. Mrs. Reena Saxena has worked under my guidance and supervision and has fulfilled the requirements for the submission of this thesis, which to my knowledge, has reached the requisite standard.

The results contained in this thesis have not been submitted, in part or in full, to any other university or institute for award of any degree or diploma.

Ak Singh
3-7-95

Dr. A. K. Singh

Department of Chemistry

Indian Institute of Technology

New Delhi-110 016

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A handwritten signature in cursive script that reads "Reena". A horizontal line is drawn underneath the signature.

(REENA SAXENA)

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Department of Chemistry

I.I.T., Delhi

ABSTRACT

Alizarin Red-S (ARS), salicylic acid (SA), Pyrocatechol Violet (PV) and 8-hydroxyquinoline-5-sulfonic acid (SOX) were coupled with Amberlite XAD-2 after its nitration, reduction of NO_2 groups to amine and diazotization of NH_2 groups. The resulting chelating resins were characterized by elemental analyses, TGA, IR spectroscopy and reflectance spectra. All these chelating resins exhibit a band characteristic of $-\text{N}=\text{N}-$ group around $1520\text{-}1535\text{ cm}^{-1}$ and their TGA curves indicate the presence of 1-4 molecules of water per repeat unit of the polymer.

The columns (Pharmacia; C10/10) packed with 1g of either of the four chelating resins have been investigated for the sorption of bivalent metal ions $\text{Zn}(\text{II})$, $\text{Cd}(\text{II})$, $\text{Ni}(\text{II})$ and $\text{Pb}(\text{II})$. The salicylic acid functionalized Amberlite XAD-2 has also been studied for enrichment of uranium(VI). Zn, Cd, Pb and Ni have been monitored by flame atomic absorption spectrometry (AAS) and uranium by pulsed laser fluorometry (PLF). The sorption was quantitative for all the four metal ions namely Zn, Cd, Ni and Pb on Amberlite XAD-2 loaded with Alizarin Red-S or Pyrocatechol Violet. Amberlite XAD-2 loaded with salicylic acid exhibited quantitative sorption for Pb and Zn whereas SOX loaded resin was found to sorb quantitatively Cd and Pb only. The pH for quantitative sorption is below 6 for all the four resins. For desorption hydrochloric acid or nitric acid having concentrations between 1 and 4 M have been found suitable. The capacity has been found to be maximum for Amberlite XAD-2 loaded with PV. For Cd, Pb, Ni and Zn the capacity values are 1272, 1360, 618 and 1406 $\mu\text{g/g}$

of resin. The optimum flow rate controlled by peristaltic pump for quantitative sorption was found to be 1-2 cm³/min for all the systems. The preconcentration factor was maximum with Amberlite XAD-2 loaded with salicylic acid (120-180). The composite AAS methods including a preconcentration step by either of these four resins have been developed for Zn, Cd, Ni and Pb. The effect of electrolytes, (NaF, NaCl, NaNO₃, Na₂SO₄ and Na₃PO₄) on the sorption of metal ions on chelating resin have been investigated. The tolerance limits are best for Amberlite XAD-2 loaded with Alizarin Red-S. Tolerances of cations commonly present in water samples on sorption of Ni, Zn, Cd, Pb and U have also been studied. The relative standard deviations have been found to be less than 8.2% in all the cases. By comparing IR spectra of chelating resins saturated with metal ions with those of free resins, it has been concluded that Alizarin Red-S loaded resin chelates through azo and quinone groups, salicylic acid loaded resin chelates through phenolic and carboxylic groups, Pyrocatechol Violet loaded resin chelates through phenolic groups and 8-Hydroxyquinoline-5-sulfonic acid loaded resin chelates through ring nitrogen and phenolic group during sorption. Amberlite XAD-2 loaded with these resins have been used to determine Zn, Cd, Ni and Pb contents in well water samples with RSD < 10%.

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