

**FUNCTIONALIZED SILICA GEL AND
DENDRITIC/HYPERBRANCHED MACROMOLECULES:
SYNTHESIS AND METAL EXTRACTION BEHAVIOUR**

By

ANUPAMA GOSWAMI

Department of Chemistry

submitted

in fulfilment of the requirements of the degree of

DOCTOR OF PHILOSOPHY

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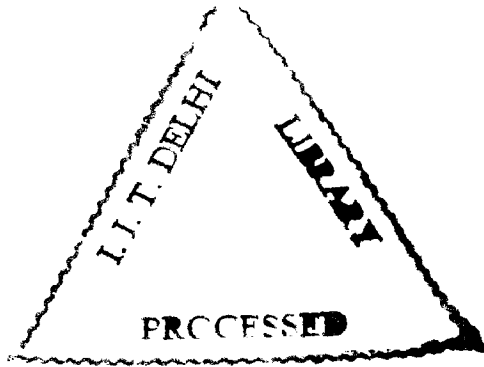


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CERTIFICATE

This is to certify that the thesis entitled, " **FUNCTIONALIZED SILICA GEL AND DENDRITIC / HYPERBRANCHED MACROMOLECULES: SYNTHESIS AND METAL EXTRACTION BEHAVIOUR**" being submitted by **Ms. Anupama Goswami** 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. Ms. Anupama Goswami has worked under my guidance and supervision, and has fulfilled the requirements for the submission of this thesis, which to my knowledge has reached requisite standard.

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

Date 21. 1. 2012



DR. AJAI K. SINGH

Professor
Department of Chemistry
Indian Institute of Technology, Delhi
New Delhi - 110 016

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ABSTRACT

Resacetophenone (2,4-dihydroxyacetophenone), 5-formyl-8-hydroxyquinoline (FHOQ_x) and 1,8-dihydroxyanthraquinone (DHAQ) have been immobilized (through >C=N- coupler) on silica gel after its modification with 3-aminopropyltriethoxy silane. The matrices were characterized by Kjeldahl method of nitrogen analysis, TGA, DRIFT and CPMAS NMR spectroscopy. Their DRIFT spectra show a characteristic band for – C=N- group apart from some of the bands of the ligands immobilized. All the matrices have been used for separation and preconcentration of Cd(II), Cu(II), Co(II), Fe(III), Ni(II), Pb(II) and Zn(II) ions, important for water quality monitoring. The optimum pH for quantitative sorption of these metal ions on resacetophenone (RATP) loaded silica gel is between 5.5 –7.5 and all the metal ions can be desorbed from the matrix with 3 M HCl or HNO₃. The sorption capacity of the matrix for the seven metal ions is in range of 57.8 - 365.0 μmol g⁻¹ of the modified silica gel. Tolerance limits for electrolytes NaNO₃, NaCl, NaBr, Na₂SO₄ and Na₃PO₄ and cations Ca(II) and Mg(II) in the sorption of these metal ions (0.1 μg ml⁻¹) are reported. The preconcentration factors are between 150-300 and t_{1/2} values < 1 min except for Cd. The 95% extraction by batch method takes ≤ 25 minutes. The limit of detection is the range 0.45-1.0 μg l⁻¹ and limit of quantification is 0.67-1.0 μg l⁻¹.

The optimum pH values for quantitative sorption of the seven metal ions on FHOQ_x loaded silica gel are between 5.0 and 7.5. Cu, Fe, Co, Ni and Cd were desorb using 2.0 M HCl or a mixture of 1.0 M HCl and 0.1 M HNO₃, while 2.0 M HNO₃ was sufficient for the desorption of the all the metal ions. A flow rate of 1.0 – 4.0 ml min⁻¹ was found suitable for both sorption and elution of metal ions. The sorption capacity

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