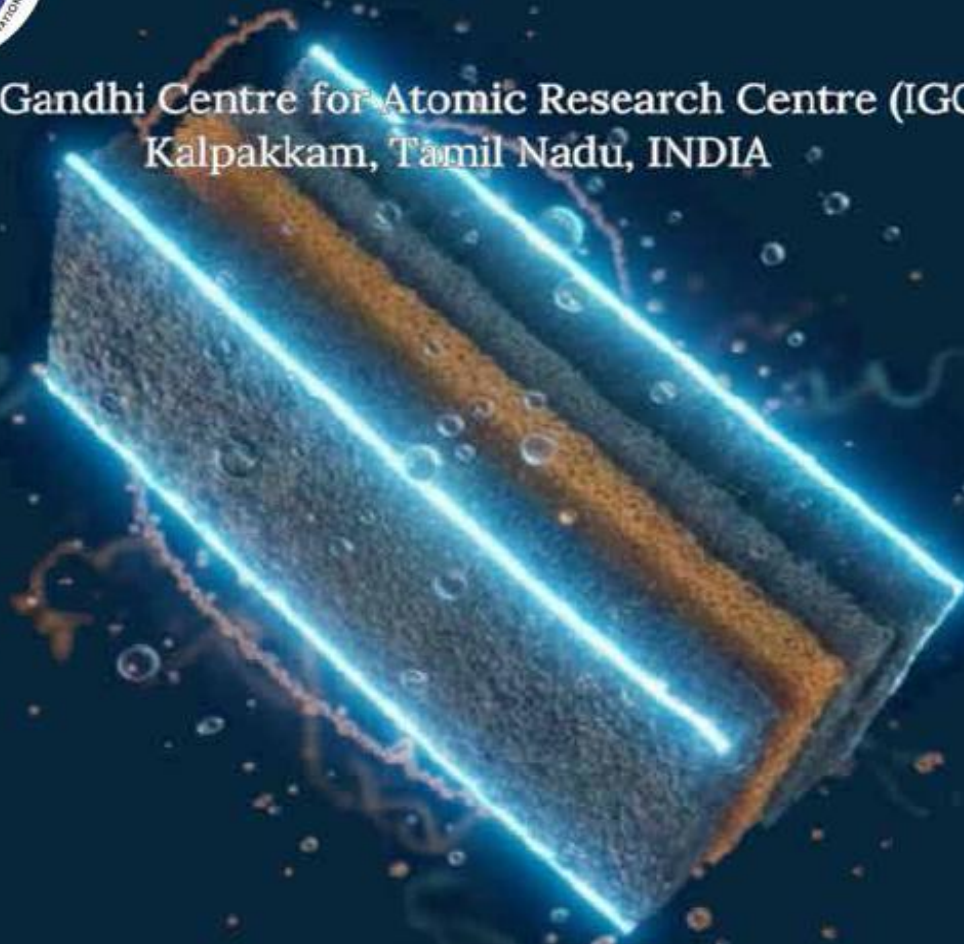




ASSET Special Bulletin on Separation Science

SESTEC -2026
February 11 - 14, 2026

Indira Gandhi Centre for Atomic Research Centre (IGCAR)
Kalpakkam, Tamil Nadu, INDIA



SESTEC -2026



Association of Separation Scientists and Technologists (ASSET)

Editors

Seraj Ansari

Arunasis Bhattacharya

Jeyakumar Subbiah



RADIATION MONITORS



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Special ASSET Bulletin
on
Separation Science



on the occasion of
SESTEC-2026



Organized by

Bhabha Atomic Research Centre, Mumbai
Indira Gandhi Centre for Atomic Research, Kalpakkam &
Association of Separation Scientists & Technologists (ASSET)

at

DAE Convention Centre, IGCAR, Kalpakkam
February 11 – 14, 2026



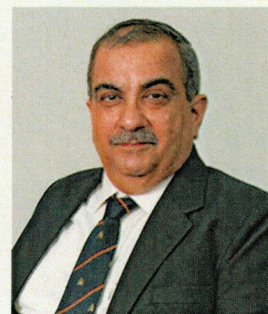
Editors

Seraj A. Ansari, Arunasis Bhattacharyya and Jeyakumar Subbiah

विवेक भसीन
Vivek Bhasin



निदेशक, भाभा परमाणु अनुसंधान केंद्र
Director, Bhabha Atomic Research Centre
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MESSAGE

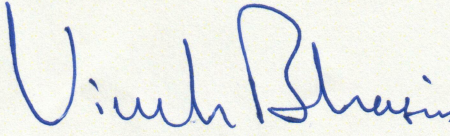
It gives me great pleasure to know that the Association of Separation Scientists and Technologists (ASSET), along with BARC and IGCAR, is organizing the 12th DAE-BRNS Symposium on *Emerging Trends in Separation Science and Technology* (SESTEC-2026) at IGCAR, Kalpakkam, during February 11-14, 2026.

Since its inception in 2004, SESTEC series of symposia has been providing excellent forum for scientists and engineers to exchange the latest developments in the field of separation science and technology, particularly in areas relevant to nuclear fuel cycle activities.

It is heartening to note that the SESTEC-2026 will be attended by a wide spectrum of participants, including young researchers and faculties from universities and research institutes, and scientists and engineers from national laboratories from India and abroad. The symposium will cover diverse themes such as Separations in the Nuclear Fuel Cycle, Radiochemical Separations, Green Separations, Membrane Science and Technology, Treatment of Industrial Effluents, Computational Chemistry in Separation Science, and Speciation Studies.

The scientific programme of SESTEC-2026 will include a keynote address, plenary lectures, invited talks, presentations by research scholars, and poster sessions spread across 14 technical sessions. I am confident that SESTEC-2026 will provide an excellent platform for fruitful deliberations and will significantly enrich the knowledge of young researchers in addressing current challenges in separation science related to industry, water treatment, and overall improvement in quality of life.

I wish SESTEC-2026 every success.


(Vivek Bhasin)

03.02.2026



श्रीकुमार जी पिल्लै
उत्कृष्ट वैज्ञानिक एवं निदेशक

SREEKUMAR G PILLAI
Outstanding Scientist & DIRECTOR



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DEPARTMENT OF ATOMIC ENERGY
INDIRA GANDHI CENTRE FOR ATOMIC RESEARCH
KALPAKKAM 603 102, TAMIL NADU, INDIA



Message

February 2, 2026

I am pleased to note that the 12th DAE-BRNS Symposium on “Emerging Trends in Separation Science and Technology (SESTEC-2026)” is being organized by the Bhabha Atomic Research Centre (BARC) in association with the Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam, and the Association of Separation Scientists and Technologists (ASSET), with full support from BRNS, at the DAE Convention Centre, Anupuram, during February 11–14, 2026. The symposium will bring together researchers from nuclear and academic institutions, faculty, and students to deliberate on advances in separation science and technology relevant to the nuclear fuel cycle, including actinide, lanthanide, and fission-product separations and speciation.

The return of SESTEC to IGCAR after a gap of 15 years is noteworthy, reaffirming Kalpakkam as a key centre of excellence in nuclear separation science. I am particularly pleased that a half-day special session is devoted to reprocessing of spent fuel, with special emphasis on fast reactor spent fuel, a topic of significant scientific and technological relevance.

As envisioned by Dr. Homi Jehangir Bhabha, India’s energy security rests on the three-stage nuclear power programme, now progressing through fast breeder reactors in Stage-II and towards thorium-based systems in Stage-III. IGCAR has a pivotal role in this national mission, with its leadership in sodium-cooled fast breeder reactor technology and the operation of the Fast Breeder Test Reactor (FBTR) using U-Pu mixed carbide fuel. Reprocessing of fast reactor fuels, closely integrated with BARC’s Kalpakkam facilities, remains central to this effort, making SESTEC-2026 a timely and relevant forum for the Kalpakkam scientific community.

Future challenges in separation science call for novel extractants, advanced process concepts, and robust technologies for Pu- and U-233-based fuels, along with safe and efficient management of high-level radioactive waste. The concept of “wealth from waste”, through recovery of valuable radioisotopes, highlights the broader societal impact of advances in this field. In parallel, pyrochemical reprocessing is gaining importance for metallic fuels envisaged for future fast reactors. IGCAR’s ongoing work in metal-fuel fabrication and pyroprocessing further enhances the relevance of discussions on both aqueous and pyrochemical routes at SESTEC-2026.

In the broader context of Amrit Kaal, the 2047 national vision, and net-zero carbon goals, progress in separation science and technology is of strategic importance for sustainable energy development, resource efficiency, and waste minimization. SESTEC-2026 thus serves as a valuable platform to strengthen India’s leadership in this domain, foster inter-institutional collaboration, and translate research outcomes into practical solutions for the nuclear fuel cycle and radioactive waste management.

I convey my best wishes for the grand success of SESTEC-2026.


(Sree Kumar G Pillai)
Director



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सत्यमेव जयते



Dr. R. B. Grover
Chairman

Government of India
Department of Atomic Energy (DAE)
Board of Research in Nuclear Sciences (BRNS)

02 February 2026

MESSAGE

I am pleased to know that the 12th DAE-BRNS Biennial Symposium on “Emerging Trends in Separation Science and Technology” (SESTEC-2026) is being organised at IGCAR, Kalpakkam, during February 11-14, 2026.

The Separation Science and Technology play a critical role in various stages of nuclear fuel cycles, viz., the recovery of uranium and thorium and their purification, retrieving fission products and fissile materials from spent fuel, and partitioning of minor actinides for safe management of nuclear waste. Besides nuclear industries, Separation Science and Technology play a key role in sectors such as pharmaceuticals, petrochemicals, water purifications, etc. Innovations in Separation Science and Technology are important to all these sectors to make the processes efficient, selective, cost-effective, environmentally benign and commercially viable.

As in any symposium, SESTEC-2026 will provide a platform to scientists and engineers working in Separation Science and Technology to interact and share their ideas. Given the importance of reprocessing spent fuel for the nuclear programme in India, I am delighted to note that a special session is dedicated to reprocessing. I hope the researchers working in this field will benefit from deliberations in the symposium and will move forward to overcome various challenges in the back-end of the nuclear fuel cycle.

I am happy to know that a large number of researchers from India and some from abroad are expected to participate in the symposium. I compliment the organisers, and I must say that this symposium will benefit scientists, researchers, and students through deliberations and possible collaborations.

I extend my best wishes for the grand success of SESTEC-2026.

(R B Grover)

From President's Desk...



Prof. Jeyakumar Subbiah
Head, Radiochemistry Division
Bhabha Atomic Research Centre
Mumbai 400 085, India
Professor, HBNI

Separation science is inevitable in every part of nuclear science and technology. It plays a pivotal role in achieving carbon-free nuclear power, which includes the separation of ores and minerals, the production of nuclear and reactor materials, the separation of isotopes, the application of radioisotopes in various sectors, such as health and agriculture, the reprocessing of spent fuels, and the management of nuclear waste.

The Association of Separation Scientists and Technologists (ASSET), a registered professional body, acts as a binding force and provides a common platform for the researchers and technologists to discuss the advancements in separation science and to take up new challenges. ASSET encouraged many scientists and engineers to get into collaborative projects.

ASSET organises a number of half-day seminars, special lectures and theme meetings on various topics in the field of separation science and technology. It also publishes the thematic bulletins regularly. ASSET supports young researchers and research scholars in carrying out short-term research projects at national institutes and DAE organisations. ASSET also organises workshops for young researchers and PG students.

ASSET co-organises the SESTEC series of symposia with the full grant from DAE-BRNS, and SESTEC provides a wonderful opportunity for the scientists, engineers, technologists, and young researchers to interact, deliberate, and share their views and identify ways to resolve their challenging issues in separation science.

On behalf of ASSET, my warm welcome to all the participants, and I wish SESTEC-2026 a resounding success.

February 04, 2026.

A handwritten signature in blue ink, appearing to read 'J. K. S.', written over a light blue horizontal line.

(Jeyakumar S)
President, ASSET

Preface

Separation Science and Technology occupies a central role in the advancement of nuclear fuel cycle chemistry, environmental remediation, resource recovery, and materials innovation. The ability to selectively isolate, recover, and purify elements of strategic importance from complex matrices continues to drive research at the interface of chemistry, materials science, and engineering. Over the years, this multidisciplinary field has evolved from conventional solvent extraction and ion exchange methods to the development of advanced functional materials, hybrid composites, and intelligent adsorbent systems tailored for specific separation challenges.

This Special ASSET Bulletin is brought out on the occasion of the Twelfth DAE-BRNS Biennial Symposium on Emerging Trends in Separation Science and Technology (SESTEC-2026), organized by Bhabha Atomic Research Centre, Mumbai, in association with the Association of Separation Scientists and Technologists (ASSET) at the DAE Convention Centre, IGCAR, Kalpakkam. SESTEC has, over the past two decades, provided a vibrant platform for scientists, technologists, and researchers to exchange ideas, present innovations, and deliberate on emerging directions in separation science relevant to nuclear, environmental, and industrial applications.

The articles compiled in this bulletin reflect the contemporary research trends that are shaping the future of separation technologies. They highlight the transition from traditional approaches to advanced materials such as graphene oxide composites, magnetic nano-adsorbents, and metal-organic frameworks (MOFs), demonstrating how rational design of functional materials can address complex challenges in the selective recovery of uranium, zirconium, palladium, and other critical metal ions from aqueous streams. Contributions also extend to applications in nuclear medicine, drinking water purification, cloud point extraction, and mass transfer modelling in advanced separation columns, illustrating the breadth and versatility of the field.

A recurring theme across these contributions is the integration of material design, surface chemistry, and process engineering to develop separation systems that are efficient, selective, environmentally benign, and scalable for real-world deployment. The work presented herein underscores the importance of interdisciplinary collaboration in addressing the evolving needs of the nuclear fuel cycle, waste management, and sustainable resource utilization.

We sincerely acknowledge the authors for their valuable contributions and the reviewers for their insightful suggestions. We also express our gratitude to ASSET, and all members of the organizing team whose efforts made both this publication and SESTEC-2026 possible. We gratefully acknowledge the invaluable support of Dr. P.G. Jaisan, Dr. Aishwarya Kar, Dr. Sumana Paul, Dr. Rubel Chakravarty, Dr. Rajesh Gujar, and all members of the Exhibition Committee.

It is our earnest hope that this bulletin will serve as a useful reference for researchers, inspire new ideas, and foster continued innovation in the fascinating and ever-expanding domain of Separation Science and Technology.

Editors

Seraj A. Ansari
A. Bhattacharyya
Jeyakumar S.

About ASSET

The ***Association of Separation Scientists and Technologists (ASSET)*** is a scientific professional body that brings together scientists and engineers engaged in the field of separation science and technology across the country. The idea of forming such an association was conceived during the concluding session of the DAE-BRNS Symposium on “*Emerging Trends in Separation Science & Technology (SESTEC)*” held in 2004, when the need for a dedicated scientific forum to promote interaction, collaboration, and capacity building in this specialized domain was strongly felt. This vision culminated in the formal registration of ASSET in 2008.

Since its inception, ASSET has been committed to advancing novel, efficient and sustainable separation technologies across diverse sectors of science and engineering, thereby contributing to national development and technological self-reliance. One of the flagship activities of the Association is the DAE-BRNS Biennial SESTEC series of symposia, which has emerged as a premier national forum for the exchange of ideas among researchers from academia, national laboratories, and industry. Additionally, ASSET has steadily expanded its academic footprint by organizing theme-based meetings, workshops, half-day seminars, ASSET Special Lectures, ASSET Day invited talks, and the prestigious Padma Shri Dr. Srikumar Banerjee Memorial Lecture. The introduction of focused workshops has been particularly impactful, providing hands-on exposure and deeper understanding of emerging techniques, instrumentation, and methodologies in separation science, especially for students and early-career researchers.

ASSET places strong emphasis on nurturing young talent and fostering inclusivity within the scientific community. The Association annually confers two awards to young researchers namely the “Thermax-ASSET Best Ph.D. Thesis Award and the Thermax-ASSET Best M.Tech. Thesis Award to recognize their outstanding research contributions. In honour of the late Dr. Priyanath Pathak, an exceptionally brilliant researcher in the field of separation science, the Dr. P.N. Pathak Memorial Award has been instituted. This award is presented biennially to a senior scientist/academician who has made exceptional and sustained contributions to separation science and technology. Furthermore, ASSET encourages research scholars by presenting Best Paper Presentation Awards during SESTEC symposia.

In its continuing efforts to support promising but financially constrained researchers, ASSET has initiated the ASSET-Prof. Manchanda Fellowship, aimed at assisting young and deserving researchers to pursue their research activities, attend scientific meetings, and enhance their professional development.

Through its symposia, workshops, lectures, awards, and fellowship programs, ASSET has evolved into a vibrant and inclusive scientific platform that not only promotes excellence in separation science and technology but also fosters mentorship, collaboration, and societal relevance. By bridging academia, research institutions, and industry, ASSET continues to strengthen the scientific ecosystem of the country. As the association looks toward the future, it remains dedicated to empowering the next generation of scientists, advancing cutting-edge research, and contributing meaningfully to national and global challenges through innovations in separation science and technology.

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**Twelfth DAE-BRNS Biennial Symposium on
Emerging Trends in Separation Science and Technology (SESTEC-2026)**
(DAE Convention Centre, Anupuram, IGCAR, Kalpakkam)
February 11 - 14, 2026
Technical Program

Wednesday, February 11, 2026		
8.30 – 9.30	Registration	
9.30-10.30	Inauguration & Key note presentation (Shri G. Sreekumar Pillai, Director, IGCAR), ASSET Award Presentation	
10.30-11.00	High Tea (Group Photo)	
11.00 -12:45	Session 1 (Chair: Dr. Y.K. Bhardwaj, Associate Director, RC&IG, BARC, Mumbai)	
	[11:00-11:35] PL-1 Radio-cesium separation strategies for nuclear fuel cycle applications (Prof. P.K. Mohapatra, Former Director, RC&IG, BARC, Mumbai)	
	[11:35-12:10] PL-10 Novel Reactions and Separations for Actinide Halides and Their Implications for the Nuclear Fuel Cycle (Prof. K.R. Czerwinski, USA)	
	[12:10- 12:45] PL-3 Radioisotope Production in Fast Breeder Test Reactor (Dr K Sundararajan, IGCAR)	
12.45-14.00	Lunch	
14.00 - 15.30	Session 2A (Chair: Dr. P.G. Jaison, RACD, BARC, Mumbai)	Session 2B (Chair: Prof. D. Prabhakaran, VIT, Vellore)
	[14:00-14:30] IT-01 Molecular-Level Understanding of Sorption and Extraction Processes: Ionic Liquids and Deep Eutectic Solvents for Advanced Separation Science (Prof. Ramesh Gardas, IITM)	[14:00-14:30] IT-04 Diffusion and Sorption behavior of tracers on Fe-montmorillonite relevant to deep geological disposal of HLW Prof. Manjanna, Rani Channamma University, Belagavi)
	[14:30-15:00] IT-02 Functionalized Polyurethane Foams - Promising Applications in Separation Processes (Prof. A. Sivaramakrishna, VIT, Vellore)	[14:30-15:00] IT-05 Radiometallic isotopes such as ⁶⁴ Cu-Copper, ⁸⁹ Zr-Zirconium, ⁴⁵ Ti-Titanium and ⁶⁸ Ga-Gallium: Production, Purification and Quality Assurance: Our Experience (Prof. Manish Dixit, IMS, Lucknow)
	[15:00-15:30] IT-14 Sea Bittern-Driven Forward Osmosis; Exploiting High Osmotic Pressure for Dewatering of Coconut Water (Prof. Parag Nemade, ICT, Mumbai)	[15:00-15:30] IT-06 Experimental and Theoretical Studies on Lanthanide and Actinide Speciation in Aqueous Environments – A Thermodynamic Prospective (Dr. Rama Mohana Rao Dumpala, BARC, Mumbai)
15:30-15:45	Tea Break	
15:45-17:45	Session 3	
	Session 3A Oral Presentations (12 papers) (Chair: Dr Anish Kumar, MMG, IGCAR)	Session 3B Oral Presentations (12 papers) (Chair: Dr Y. V. Nanchariah, BARCF, IGCAR)
	A-3, A-4, A-11, A-12, A-16, A-19, A-22, G-1, G-7, G-11, H-3, H-4	B-1, B-4, B-5, B-6, C-1, C-2, D-1, D-2, D-3, E-2, E-3, E-4
17:45-18:30	ASSET Award Lectures	
18:30 onward	Dinner	
Thursday, February 12, 2026		
9:30-11:10	Session 4 Special session on Reprocessing of Fuels (Chair: Dr. Rajesh Ganesan, MC&MFCG, IGCAR)	
	[9:30-10:05] PL-4 Separation Science and Technology at Backend of Fuel Cycle: Current Status and Way Forward (Dr. G. Sugilal, Director, NRG, BARC)	
	[10:05-10:40] PL-5 Advanced process flow-sheet for fast reactor fuel reprocessing (Dr. K.A. Venkatesan, IGCAR)	
	[10:40- 11:10] IT-07 Pyrochemical Reprocessing Technology for Metal Fuel Fast Reactors: Development Status and Deployment Plans (Dr. S.P. Ruhela, IGCAR)	
11:10-11:15	Tea Break	

11:15-13:05	Session 5 (Chair: Dr. S. Kannan, Former Director, RC&IG, BARC, Mumbai)	
	[11:15-11:45] IT-08 Recovery Current Strategies for Americium Recovery from High-Level Waste: International Scenario (Dr. S.A. Ansari, BARC, Mumbai)	
	[11:45-12:15] IT-9 Advancements in Separation Science and Technology in Nuclear Fuel Cycle and its Implications on Sustainability and Circularity of the Nuclear Power and Waste Management Program (Dr. G. Suneel, NRB, Kalpakkam)	
	[12:15-12:35] Technical Invited Talk New trends in ICP-OES (Mr R. Karthikeyan, Technical Manager, Advance Scientific Equipments Pvt Ltd)	
	[12:35-13:05] IT-10 Process Chemistry of Fast Reactor Fuel Reprocessing and Recovery of Valuables from Fast Reactor Waste (Dr K Dhamodharan, RpG, IGCAR)	
13.05-14.00	Lunch	
14:00-16:00	Session 6	
	Poster presentation & Floating Tea A1, A-2, A-5, A-6, A-7, A-8, A-9, A-10, A-13, A-14, A-15, A-17, A-18, A-20, A-21, A-23, A-24, A-26, A-27, A-29, A-30, A-31, A-33, A-37, A-39, A-40, B-2, B-3, B-7, B-10, B-11, B-12, C-4, D-4, D-10, D-11, D-13, D-14, D-15, E-1, E-5, E-9, E-10, E-11, E-13, F-1, F-2, F-3, G-2, G-3, G-4, G-5, G-6, G-8, G-9, G-10, H-1, H-2, H-6, H-8	
	Session 7	
	Session 7A : Oral Presentations (12 papers) (Chair: A. Suresh, Former Head, FChD, IGCAR)	Session 7B: Oral Presentations (12 papers) (Chair: Geo Matthews, RpG, IGCAR)
16:00-18:00	A-25, A-28, A-32, A-34, A-35, A-38, G-12, G-13, G-14, G-15, H-5, H-7	B-8, B-9, B-14, C-3, C-6, D-5, D-6, D-7, E-6, E-7, E-8, F-4
18:00 onward	Floating Tea/ Cultural Program and Dinner	
Friday, February 13, 2026		
9:30-11.10	Session 8 (Chair: Prof. V.K. Manchanda, Former Head, RCD, BARC)	
	[9:30-10:05] PL-6 The vital role of radioisotope separation science in nuclear medicine (Dr. Madhav B. Mallia BARC, Mumbai)	
	[10:05-10:40] PL-7 Engineering of Solvent Extraction Process Development (Shri K.T. Shenoy, Former Director, ChEG, BARC)	
	[10:40-10:50] Technical Talk-1	
	[10:50-11:00] Technical Talk-2	
	[11:00-11:10] Technical Talk-3	
11:10-11:30	Tea Break	
	Session 9	
11:30-13:00	Session 9A (Chair: K. Rajan, Former Director, RpG, IGCAR)	Session 9B (Chair: J. Kodandaraman, RpG, IGCAR)
	[11:30-12:00] IT-11 Processing of Uranium Ore at UCIL Mills of India Dr. J. Dinesh Kannan (UCIL)	[11:30-12:00] IT-03 Extraction of Critical Minerals - Breaking the Bottleneck (Dr. Benadict Rakesh, CSIR-IMMT, Bhubaneswar)
	[12:00-12:30] IT-12 From Ores to Wastes: Integrated Strategies for Sustainable Recovery of Rare Earth Elements (Prof. D.K. Singh, IIT(ISM), Dhanbad)	[12:00-12:30] IT-15 Density Functional Theory Studies on the Separation and Recovery of Lanthanides and Actinides (Dr. G. Gopakumar, IGCAR)
	[12:30-13:00] IT-13 Innovative Approaches for Managing Complex Radioactive and Mixed Wastes (Dr. S. Sriram, Canada)	[12:30-13:00] IT-16 Diverse applications of Polyoxometalates (POMs) in the field of Actinides (Dr. B. Mahanty, BARC, Mumbai)
13.00-14.00	Lunch	
	Poster presentation & Floating Tea A-41, A-43, A-45, A-46, A-47, A-48, A-49, A-52, A-55, A-56, A-57, A-58, A-59, A-60, A-61, A-62, A-63, A-64, A-65, A-66, A-67, A-69, A-70, A-71, A-72, B-13, B-18, B-19, B-20, B-21, B-22, C-5, C-7, D-16, D-17, D-21, D-22, D-23, E-15, E-17, E-18, E-20, E-21, E-22, E-23, F-5, F-6, F-7, G-16, G-19, G-20, G-21, G-22, G-23, G-24, G-26, H-9, H-11, H-12, H-13	
	Session 10	
	Session 10A: Oral Presentations (12 papers) (Chair: Dr. R. Gopalakrishnan)	Session 10B: Oral Presentations (12 papers) (Chair: Prof. P. Sri Ranganathan)
16:00-18:00	A-42, A-44, A-50, A-51, A-53, A-54, A-68, G-17, G-18, G-25, H-10	B-15, B-16, B-17, C-8, C-9, D-8, D-9, D-18, D-19, E-12, E-14, E-16
19:30	Dinner	

Saturday, February 14, 2026		
(9:30-11:15)	Session 11 (Chair: Prof. N. Sivaraman, Former Director, MC&MFCG)	
	[9:30-10:05] PL-8 Application of Ion Exchange Chromatography to establish SI Traceability in Certified Reference Materials (CRMs) of Bio-molecules (Dr. K. Dash, Head, NCCCM, Hyderabad)	
	[10:05-10:40] PL-9 Polymer inclusion membranes and beads in environmentally friendly chemical separation (Prof. S.D. Kolev, Australia)	
	[10:40-11:15] PL-2 Separation Technologies in advanced Nuclear Fuel Cycle (Shri Kalyan Bhanja, AD, ChEG, BARC)	
11:15-11:30	Tea Break	
11:30-13:20	Session 12	
	Session 12A (Chair: T. V. Prabhu, MC&MFCG)	Session 12B (Chair: S. Chandramouli, FRTG)
	[11:30-12:00] IT-17 Advanced Functionalized Porous Monolithic Materials: Enabling Next-Generation Separation and Sensing Technologies (Prof. Akhila Maheswari, VIT, Vellore)	[11:30-11:50] SIT-01 Application of cloud point extraction (CPE) in various fields of the nuclear industry (Dr. Abhijit Saha, BARC)
	[12:00-12:30] IT-18 Synthesis and Evaluation of Novel Functionalized Metal-organic Frameworks for the Recovery of Metal Ions (Dr. B. Sreenivasulu, IGCAR)	[11:50-12:10] SIT-02 Speciation of f-elements for sustainable growth of nuclear and renewable energy (Dr. Ashutosh Srivastava, BARC, Mumbai)
	[12:30-13:00] IT-19 Recent advancement in the Chemical Characterization of (U, Pu)O₂ MOX fuel (Dr. Anoop Kelkar, NRB, Tarapur)	[12:10-12:30] SIT-03 Alternate Ligand – Solvent System For PUREX Process (Dr. Arya Das, NRB, Mumbai)
		[12:30-12:50] SIT-04 Role of DFRP in closing the fast reactor fuel cycle (Shri. R Velmurugan, RPG, IGCAR)
13.20-14.15	Lunch	
	Session 13	
14:15-14:35	Overview on BRNS Activities Shri D. Mathur, BRNS, Mumbai	
14:35-15:00	Panel Discussion	
15:00-16:00	Concluding Session (Chief Guest: S. Satyakumar, CE, HWB)	

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Probe Embedded Porous Inorganic / Organic / Hybrid Monolithic Scaffolds for the Solid-State Naked-Eye Detection and Recovery of Toxic Heavy Metal Ions: A Mini-Review

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Summary

The rapid and reliable detection of heavy metal ions is of critical importance due to their environmental persistence, bioaccumulation, and associated health risks. In recent years, solid-state colorimetric sensors have emerged as promising alternatives to conventional solution-phase techniques owing to their simplicity, portability, and on-site applicability. Among solid platforms, monolithic and porous materials have attracted significant attention due to their high surface area, interconnected pore architecture, and ease of functionalization with chromoionophoric probes. This mini-review summarizes recent advances in solid-state colorimetric sensing of heavy metal ions using diverse monolithic and porous platforms deployed by our research group, including silica monoliths, organic polymer monoliths, hybrid / hydrolyzed monoliths, and metal-organic frameworks. Emphasizing material design strategies, probe immobilization methods, sensing mechanisms, and analytical performance. The advantages and limitations of various porous materials are discussed, with particular emphasis on their suitability for selective and sensitive metal-ion detection.

Introduction

Heavy metal ion contamination poses a serious threat to environmental safety and human health owing to its non-biodegradable nature, long-term persistence, and tendency to bioaccumulate [1–3]. Toxic metal ions such as Cu^{2+} , Co^{2+} , Fe^{3+} , Hg^{2+} , Pb^{2+} , Cd^{2+} , $\text{Cr}^{3+/6+}$, As^{3+} , and UO_2^{2+} are commonly released into aquatic systems through industrial effluents, mining activities, nuclear processes, and agricultural runoff, posing serious risks to ecosystems and human health[4,5]. Consequently, the development of rapid, reliable, and cost-effective sensing strategies for heavy metal ions remains a critical challenge in environmental and analytical chemistry. Conventional

analytical techniques, including atomic absorption spectroscopy (AAS) [6], inductively coupled plasma mass spectrometry (ICP-MS), and inductively coupled plasma optical emission spectroscopy (ICP-OES) [7,8], offer a high sensitivity and accuracy for metal ion analysis. However, these techniques require expensive instrumentation, skilled personnel, and extensive sample preparation, thereby limiting their applicability for rapid, on-site detection.

In contrast, colorimetric sensing has emerged as an ideal alternative due to its simplicity, low cost, fast response, and the ability to enable visual detection without sophisticated equipment [9]. Organic solvent (solution)-phase colorimetric sensors have been widely explored for heavy metal ion detection; however, their practical application is often constrained by drawbacks such as probe leaching, limited reusability, instability under varying environmental conditions, and interference from complex sample matrices [10–12]. To address these limitations, solid-state colorimetric sensors have gained increasing attention. Immobilization of chromoionophoric probes onto a solid support enhances sensor stability, reusability, and portability, while enabling direct application in real-world samples [13,14].

Among various solid supports, monolithic and porous materials have emerged as up-and-coming platforms for solid-state colorimetric sensing. Their high surface area, interconnected pore networks, and tunable surface chemistry facilitate efficient probe immobilization and rapid diffusion of analyte ions [15]. Silica monoliths were among the first porous materials explored due to their well-defined pore structure and ease of surface functionalization via silanol chemistry [16,17]. However, limitations related to mechanical fragility and chemical stability under extreme pH conditions prompted the development of alternative platforms. So, research on organic polymer monoliths has emerged as a robust alternative to silica supports due to their superior chemical stability, mechanical flexibility, and tunable porosity. Controlled selection of monomer and crosslinker enables high chromoionophore loading, reduced probe leaching, and enhanced durability, making polymer monoliths well-suited for reusable solid-state colorimetric sensing in real-sample environments [18,19]. Further advances led to the emergence of hybrid organic-inorganic monoliths, which combine the rigidity of inorganic frameworks with the flexibility and functional diversity of organic polymers, thereby improving mechanical stability and sensing performance [20,21]. Hydrolyzed hybrid monoliths represent a further refinement, in which controlled hydrolysis generates additional surface hydroxyl groups, thereby enhancing probe anchoring efficiency and sensitivity toward target metal ions [22–24]. More recently,

metal-organic frameworks (MOFs) have also been explored as advanced porous platforms owing to their crystalline nature, high surface area, and tunable pore environments, which enable selective host-guest interactions and signal amplification in colorimetric sensing applications.

Although substantial progress has been made in the development of solid-state colorimetric sensors, a systematic comparison of monolithic and porous platforms with respect to material architecture, probe integration, and sensing efficiency remains lacking. This mini-review surveys recent developments in solid-state colorimetric detection of heavy metal ions using a range of monolithic and porous supports. Particular attention is given to the influence of material composition and surface chemistry on chromoionophore immobilization, signal transduction mechanisms, and analytical performance. Silica monoliths, organic polymer monoliths, hybrid and hydrolyzed hybrid monoliths, and metal-organic framework-based platforms are comparatively evaluated to highlight their distinct advantages and inherent limitations. Fig. 1 provides a general schematic illustration of sensor material fabrication using various porous materials as templates for the quantification of toxic heavy metal ions and naked-eye colorimetric detection. Finally, solid-state sensor materials effectively address key challenges related to stability, selectivity, and practical implementation.

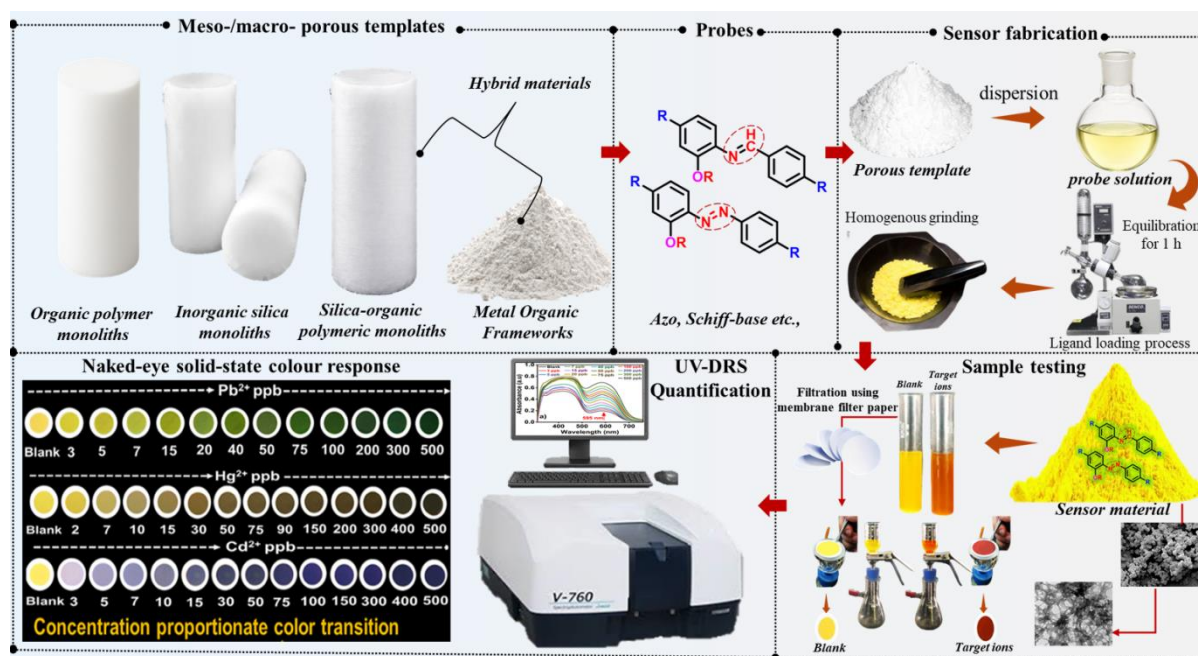


Fig. 1. Schematic representation of the fabrication of solid-state colorimetric sensors for metal ion sensing.

Porous Monolithic Scaffolds

Inorganic Monolith: Silica monolith

The microemulsion-based sol-gel process was employed to fabricate a silica monolith template; the tri-block copolymer surfactant served as the template to organize the silica precursor into a structured framework. Controlled pore size and uniform pore distribution throughout the silica monolith enable homogeneous dispersion of probe molecules, facilitating faster access of target analytes to the probe binding sites. Mesoporous silica monoliths exhibit enhanced surface area and porosity, thereby supporting the development of efficient solid-state sensor systems. The inherent chemical stability of mesoporous silica monoliths ensures durability, and the frozen encapsulation of the probe moiety enhances ion selectivity and improves sensitivity.

Kongasseri et al. synthesized a mesoporous silica monolith (MSM) using F127 as a structure-directing agent (SDA), which exhibited a honeycomb-like mesoporous network. SEM analysis confirmed a continuous interconnected structure, and the rough surface enabled efficient immobilization of the HMTAR probe. The resulting HMTAR-anchored MSM functioned as a dual solid-state colorimetric sensor for Cd^{2+} and Hg^{2+} , showing visible color changes from pale orange to dark red (Cd^{2+}) and pale orange to purple (Hg^{2+}), within ≤ 10 min, with LODs of 2.67 and 2.90 ppb for Cd^{2+} and Hg^{2+} , respectively [26]. Similarly, Madhesan et al. reported a silica-based template (MSM-P123-TiPB) immobilizing a TDQ chromoionophore for Co^{2+} detection, exhibiting a rapid color transition from light orange to bright red within ≤ 4 min and a lower LOD of 0.12 pp [27]. Furthermore, Kongasseri et al. fabricated PEO-MSM and F108-MSM via a sol-gel process using different surfactants, both of which were uniformly loaded with BOICB probe molecules. Although PEO-MSM ($254.85 \text{ m}^2/\text{g}$, $0.27 \text{ cm}^3/\text{g}$, and 3.58 nm) and F108-MSM ($370.70 \text{ m}^2/\text{g}$, $0.32 \text{ cm}^3/\text{g}$, and 2.12 nm) exhibited comparable textural properties, F108-MSM displayed a highly ordered 3D-caged cubic mesostructured, while PEO-MSM showed a disordered, worm-like morphology. These structural differences influenced the selective sensing performance of Hg^{2+} . They led to a visible color transition from light pink to deep violet, with LODs of 0.61 and 0.22 ppb for PEO-MSM- and F108-MSM sensors, respectively [28]. The various chromoionophores immobilized on the silica monolith support are depicted in Fig. 2.

In another study, Veedu et al. synthesized a mesoporous polymer silica template (MPSM) using F127, TEOS, and TiPB, which exhibited a dense interconnected network with high surface area ($582 \text{ m}^2/\text{g}$), pore volume ($0.52 \text{ cm}^3/\text{g}$), and pore diameter (354 nm) as shown in Fig. 3 (a-j).

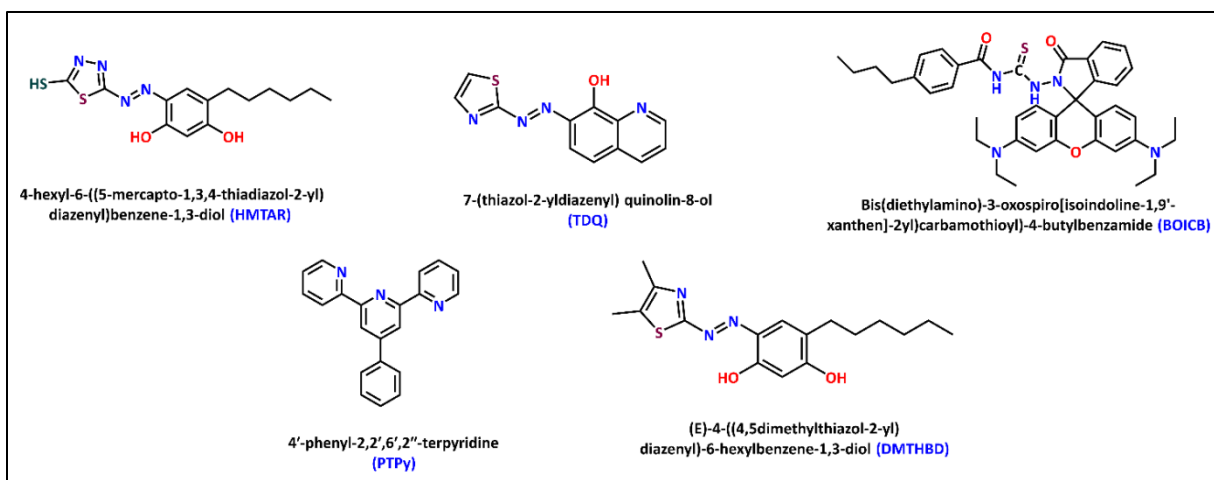


Fig. 2. List of chromoionophoric probes for immobilization onto a silica monolith.

The PTPy-loaded MPSM enabled selective detection of Co^{2+} and Fe^{3+} , showing a color transition from pastel light lavender to dark orange (Co^{2+}) and violet (Fe^{3+}), and exhibits a lower LODs of 0.5 and 0.3 ppb for Co^{2+} and Fe^{3+} , as shown in Fig. 3 (k-n) [29]. Likewise, Veedu et al. developed an eco-friendly solid-state opto-chemosensor by embedding a DMTHBD azo-receptor within a mesoporous honeycomb-structured silica framework (MHSF). The MHSF exhibited a high surface area ($745 \text{ m}^2/\text{g}$), pore volume ($1.10 \text{ cm}^3/\text{g}$), and pore diameter ($29.76 \text{ \AA}$), enabling rapid detection of Cd^{2+} upon probe embedding. The DMTHBD@MHSF sensor exhibited a rapid color change from salmon pink to intense violet within $\leq 50 \text{ s}$ and a superior LOD of 0.15 ppb.

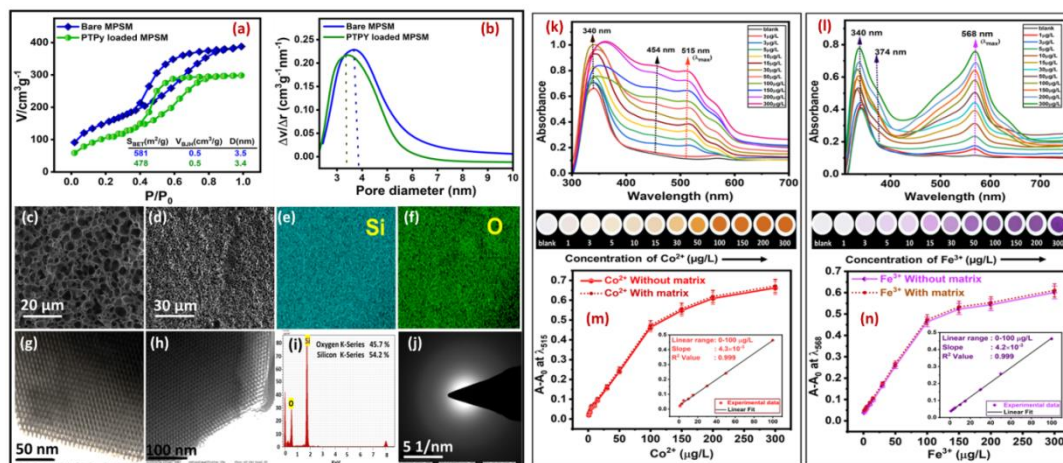


Fig. 3. (a) BET and (b) BJH pore size distribution data for bare MPSM and sensor material, (c&d) FE-SEM image of MPSM, (e&f) elemental mapping of MPSM, (g&h) HR-TEM photomicrographs of MPSM, (i)EDAX data,(j) SAED pattern, (k&l) UV-Vis DRS signal response and incremental visual color transition for Co^{2+} and Fe^{3+} , and (m&n) Calibration plot (inlet plot indicates the linear response range) of Co^{2+} and Fe^{3+} .

Organic Monolith

Although silica-based monoliths are well recognized for their high selectivity toward target metal ions, their sensing performance often deteriorates under acidic conditions due to disruption of the ordered porous framework, leading to compromised structural integrity and reduced detection reliability. To overcome the limitations of inorganic monoliths, porous organic polymer monoliths have emerged as a promising alternative, offering enhanced chemical stability, high surface area, and well-defined porosity. Owing to their flexible organic backbone and rich functionalization capability, polymer monoliths enable stronger interactions with molecular probes, thereby improving target-specific recognition, selectivity, and sensing efficiency. Furthermore, their robust framework supports effective regeneration and reusability, making them attractive platforms for solid-state sensing applications.

The macro- and mesoporous dual-combination network of the organic polymer monolith is formed via a thermally assisted free-radical bulk polymerization mechanism. These reactions are initiated by a radical initiator, which activates the allylic moieties of the monomer units. The exact temperature required for polymerization is perhaps the most crucial parameter influencing polymerization kinetics, as it induces rapid initiator decomposition, thereby facilitating the formation of numerous entangled growing polymer chains. Moreover, the porogen molecules facilitate the formation of uniform pores. The various chromoionophores immobilized on the organic polymer monolith are depicted in Fig. 4.

Sompalli et al. developed a solid-state sensor material using a structurally organized polymer monolith as a template for the trace-level detection of Cu^{2+} . Initially, three organic polymer monoliths were synthesized using the same monomer (vinyl pyridine) and crosslinker (EGDMA), but with different porogenic solvents, namely dioxane, cyclohexanol, and DMSO. Among them, the organic polymer monolith synthesized in 1,4-dioxane exhibited higher surface area and pore properties. The resulting bare polymer monoliths were fabricated using three chelating probes, namely BNHCB, TMHCA, and BDMPD, yielding LIP-1, LIP-2, and LIP-3. The bare monolith exhibits a continuous, densely packed porous framework with a rough, evenly distributed surface. Hence, the fabricated sensor materials exhibit lower LODs of 0.56, 0.30, and 0.12 ppb for LIP-1, LIP-2, and LIP-3, respectively [31]. Likewise, Sivaraman et al. constructed two porous organic polymer monoliths, namely poly(NAT-EGDMA) and poly(NAT- co-TM),

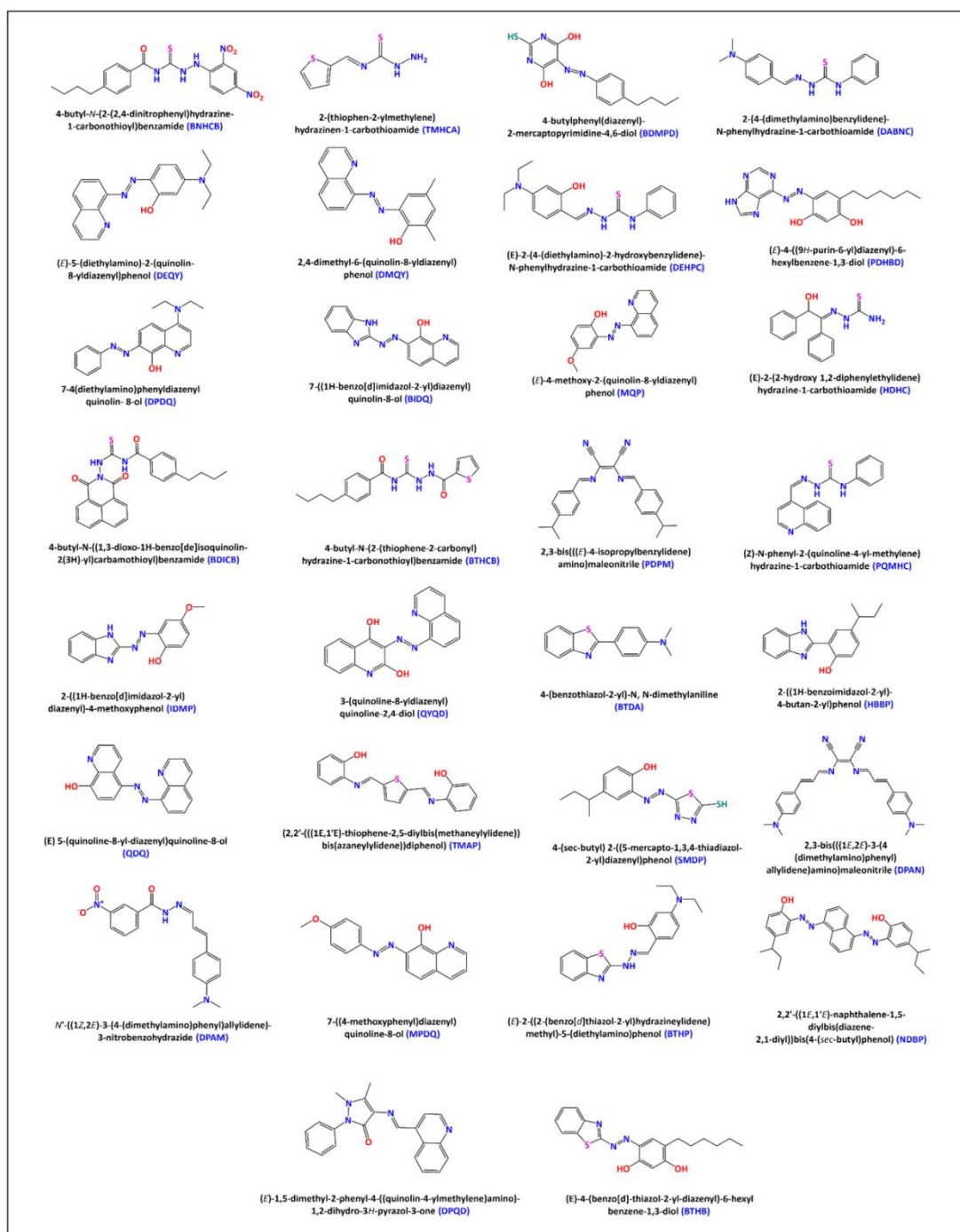


Fig. 4. List of chromoionophoric probes for immobilization onto an organic monolith.

with the same functional monomer, allylthiourea (monomer), but with different crosslinkers and porogenic solvent mixture. The surface topography of the polymer templates exhibits a single blocky flaked-aggregation with a well-defined porous network throughout the template, making it an effective anchor for probe molecules. Initially, the poly(NAT-EGDMA)

was impregnated with DABNC sensor material targeting Cu^{2+} , which exhibits a visible color transition from dull white to sap green, with an LOD of 0.15 ppb [32]. In contrast, poly(NAT-co-TM) was well-embedded with two probe molecules (DEQY & DMQY), yielding poly(NAT-co-TM)DEQY and poly(NAT-co-TM)DMQY targeting Co^{2+} . The visible color transition from light orange to dark pink for poly(NAT-co-TM)DEQY and from light sandal to dark brown for poly(NAT-co-TM)DMQY, with lower LODs of 0.14 and 0.18 ppb, respectively [33].

Srinivasan et al. proposed a solid-state sensor for recognizing Co^{2+} using a bimodal meso-/macro-porous polymer monolith as a template, which was well embedded with DEHPC probe molecules. The fabricated template exhibits a multi-layer cage-like porous network with a rigid structural framework and an intertwined (bimodal) porous structure. The sensor material exhibits selective detection of Co^{2+} , with a visible color change from pale yellow to dark green, with a LOD of 0.2 ppb [34]. In a later study, Kuppusamy et al. designed a structurally engineered porous polymer monolithic template for the homogeneous anchoring of the PDHBD receptor. The uniformly anchored probes on a bimodal macro-/mesoporous polymer monolith template (AM-co-EDGMA) perform as a solid-state optical sensor for the selective sensing/recovery of ultra-trace Co^{2+} with a visible color transition from pale orange to deep red, with a LOD of 0.3 ppb [35].

Madhesan et al. studied different porous organic polymer (POP) templates using four monomers, such as DMA, BMA, MBA, and AA, which were polymerized using AIBN as initiator with various crosslinkers, such as TMPTA, EDGMA, and PETA, and a suitable porogen. Among all the POPs, poly(DMA-co-TMPTA) exhibits remarkable structural properties, with a surface area of $455.24 \text{ m}^2/\text{g}$, a pore volume of $1.0 \text{ cm}^3/\text{g}$, and a pore diameter of 10.65 nm. The receptor (DPDQ) impregnated poly(DMA-co-TMPTA)-DPDQ forms a stable complex with Hg^{2+} , resulting in a notable color change from pale orange to bright reddish brown with a lower LOD of 0.1 ppb [36].

Similarly, Kuppusamy et al. developed a porous monolithic polymer using GMA (monomer), EGDMA (crosslinker), and 1:2 of dry DMF and 2-dodecanol. The chromoionophoric probe BIDQ was uniformly distributed on the bare polymer monolith, yielding a BIDQ sensor that targets Hg^{2+} . The bare monolith exhibits a continuous, interconnected meso-/macro-porous network. Thus, the bimodal pores enable the voluminous dispersion of BIDQ probe molecules. The BIDQ sensor shows a visible color transition from

pale pink to deep violet with a LOD of 0.2 ppb [37]. Likewise, P. Srinivasan et al. reported a solid-state sensor for ultra-trace, naked-eye colorimetric detection of Cd^{2+} . The probe molecules (MQP) were well distributed onto the bimodal organic polymer monolith template. The template, poly(VPD-co-TRIM), was synthesized by dissolving VPD and TRIM in a 15 mL porogenic solvent (10 mL of DMF, 2 mL of 1,3 butanediol, and 3 mL of dodecanol). The template's surface morphology comprises spherical, interweaved nano-beads with intermingled pores. The MQP sensor shows a visible color transition from pale pink to red wine with a lower LOD of 0.32 ppb.

Krishna Kumar et al. fabricated a solid-state optical sensor using a chromoionophoric probe molecule (HDHC) and a tailor-made porous organic polymer monolith (poly(GMA-co-PETMA)) for trace-level detection of Cu^{2+} . The porous organic polymer monolith exhibited well-ordered spherical macropores and an evenly interconnected mesoporous network, enabling uniform and high adsorption capacity for the HDHC receptors. The poly(GMA-co-PETMA)HDHC sensor material shows a visible color transition from light yellow (blank) to brownish green (HDHC- Cu^{2+}), with a lower LOD of 0.68 ppb [39]. On a similar concept, P. Srinivasan et al. also fabricated a sensor material for detecting Cu^{2+} . The solid-state template was prepared by dissolving V-2P (1 mmol, monomer) and EGDMA (10 mmol, crosslinker) in dry DMF (porogenic solvent) to generate the MMPM template. For the colorimetric detection of Cu^{2+} , two chromogenic probes (BDICB & BTHCB) were uniformly embedded within the MMPM template, yielding sensors I and II. Due to the high surface area and voluminous porosity that enabled uniform probe anchoring, the sensors exhibit a kinetic response of ≤ 1 min for the naked-eye color transition from the colourless state to apple green and greyish black, with LODs of 0.11 & 0.14 ppb for BDICB- & BTHCB-anchored sensor materials, respectively [40].

Followed by Gigi et al., developed a solid-state opto-sensor for the selective detection of ultra-trace Hg^{2+} . The developed chemosensor was fabricated by directly impregnating a chromoionophoric probe into the structurally tailored porous polymer monolithic framework (poly(AM-co-EGDMA)). The template's surface morphology exhibits a rough, monolithic appearance with a highly ordered, heterogeneous distribution and an "inter-globular" development; such characteristics imply that the available mesopores in the polymer were accessible to the probe moieties. Thus, the poly(AM-co-EGDMA)-PDPM shows a visible color transition from dull white to dijon yellow upon addition of Hg^{2+} with LOD of 0.22 ppb [41]. Accordingly, Anju et. al. synthesized a bimodal macro-/mesopore-structured polymer monolith

(poly(AAm-co-EGDMA)), which provides voluminous, uniform anchoring of probe molecules (PQMHC), yielding a sensor material named poly(AAm-co-EGDMA)PQMHC. The surface morphology reveals a coarse solid texture with a uniform network of macro- and mesopores. The sensor shows a visible color transition from soft ivory (blank) to intense orange (PQMHC-Hg²⁺), with a LOD of 0.3 ppb and response kinetics within ≤ 30 s [42].

Similarly, Srinivasan et al. developed a selective, sensitive, solid-state colorimetric method for the detection and recovery of Pb²⁺, Hg²⁺, and Cd²⁺. For solid-state sensing, an indigenously synthesized polymer monolith, i.e., poly(VAn-EGDMA), in-built with a high surface area and an interlinked meso-/macro-pore network, serves as a template for the voluminous and uniform decoration of chromoionophoric probe molecules(IDMP), onto the template. The IDMP@poly(VAn-EGDMA) sensor exhibits a visible color transition from canary yellow (blank) to dark denim blue, brownish green, and lapis blue, with lower LODs of 0.44, 0.56, and 0.52 ppb for Pb²⁺, Hg²⁺, and Cd²⁺-complexed sensors, respectively [43]. Likewise, S. Sivaraman et al. demonstrated a solid-state sensor model for the selective detection of Cd²⁺. The poly(ATU-co-EGD) exhibits ordered, interlinked, flower-like network domains with a solid, rough-surface topology and well-defined pores, thereby facilitating the adherence of QYQD probes. The developed sensor offers a solid-state color transition from yellow to dark meron for a Cd²⁺-complexed sensor material. It also exhibits a lower LOD value of 0.31 ppb [44].

Veedu et al. fabricated a novel chromogenic material for the effective naked-eye detection of Hg²⁺. The poly(GMA-co-EGDMA) exhibits a structurally intertwined meso-/macro-polymer template impregnated with a chromogenic ion receptor (BTDA).The Hg²⁺-complexed sensor material exhibits a color transition from pastel yellow to olive within ≤ 40 s, with a detection limit of 0.29 ppb [45]. On the other hand, Sivaraman et al. reported a tailor-made solid-state optical sensor achieved by controlled immobilization of an amphiphilic HBBP probe onto a dual-interwoven macro-/mesoporous polymer monolithic scaffold for selective ocular sensing of ultra-trace Pb²⁺, Hg²⁺, and Cd²⁺, respectively, as depicted in Fig. 5(a-r). As shown in Fig. 5(s-y), the poly(LMC-co-TMP)HBBP sensor material exhibits a visible color transition from yellow (blank) to leafy green, peanut brown, and dark blue upon complexation with Pb²⁺, Hg²⁺, and Cd²⁺. The detection limit values are 0.46, 0.52, and 0.41 ppb for Pb²⁺, Hg²⁺, and Cd²⁺, respectively [46].

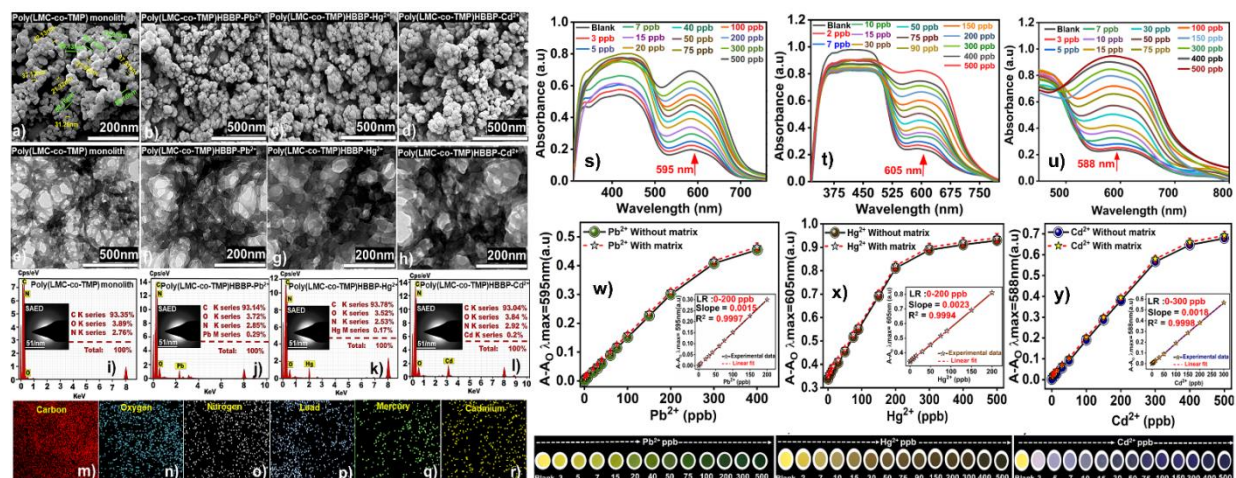


Fig 5. (a-d) FE-SEM and (e-h) HR-TEM micrographs of poly(LMC-co-TMP) monolith and corresponding $\text{Pb}^{2+}/\text{Hg}^{2+}/\text{Cd}^{2+}$ complexed HBBP sensor, (i-l) EDAX and (inlet) SAED patterns of poly(LMC-co-TMP) monolith and $\text{Pb}^{2+}/\text{Hg}^{2+}/\text{Cd}^{2+}$ complexed HBBP sensor, (m-r) elemental mapping of $\text{Pb}^{2+}/\text{Hg}^{2+}/\text{Cd}^{2+}$ complexed poly(LMC-co-TMP)HBBP sensor, (s-u) UV-Vis DRS signal response and incremental visual color transition for $\text{Pb}^{2+}/\text{Hg}^{2+}/\text{Cd}^{2+}$ complexed HBBP sensor, and (w-y) Calibration plot (inlet plot indicates the linear response range) of $\text{Pb}^{2+}/\text{Hg}^{2+}/\text{Cd}^{2+}$ complexed HBBP sensor.

Krishna Kumar et al. developed an optical on-site sensor targeting Cd^{2+} in aqueous samples. The bimodal macro/mesoporous poly(TFMAA-co-TMPTA) matrix was uniformly encapsulated with a chromophoric ligand (QDQ). The template exhibits a well-ordered, dense accretion morphology with an intertwined porous network, which maximizes the immobilization of QDQ molecules within the monolith. The Cd^{2+} -complexed sensor material shows a visible color transition from light orange to brick red, with a lower LOD of 0.53 ppb [47]. Likewise, Veedu et al. synthesized an aqua-compatible solid-state chromatic sensor for the selective detection of Cu^{2+} . The poly(2VP-co-DVB)TMAP sensor material was fabricated using a macro/mesoporous organic polymer monolith template ((2VP-co-DVB)TMAP), which facilitates homogeneous dispersion of TMAP probe molecules. Ion sensing is accompanied by a discernible color transition from vivid yellow to red, with a detection limit of 0.20 ppb [48].

Srinivasan et al. developed a solid-state visual colorimetric sensor for selective and ultra-sensitive detection of Cd^{2+} . The poly(AMST-co-TRIM) exhibits a dual-entwined porous polymer monolith imbued with SMDP probe molecules. The poly(AMST-co-TRIM)SMDP sensor material exhibits visible color shifts from light yellow to dark green, with a detection limit of 0.28 ppb [49]. With this line, Veedu et al. developed an ultra-portable azomethine-infused,

structurally interwoven polymer monolith as a solid-state chromatic sensor for the quantitative, naked-eye detection of ultra-trace Cu^{2+} . The DPAN and DPAM chromogenic probes were immobilized onto the multifaceted, cave-like pores in the disordered monolithic network of poly(4VP-co-EGDMA), yielding two sensor materials. The poly(4VP-co-EGDMA)DPAN sensor exhibited a rapid colorimetric transformation from canary yellow (blank) to vivid orange (DPAN-Cu^{2+}), with LOD of 0.11 ppb. In contrast, the poly(4VP-co-EGDMA)DPAM sensor showed a hue shift from amber yellow (blank) to rust brown (DPAM-Cu^{2+}), with an LOD value of 0.13 ppb [50].

Srinivasan et al. fabricated a solid-state, naked-eye sensor using a custom-made cross-coupled meso-/macroporous poly(VPy-co-TM) organic polymer monolith framework, infused with an MPDQ receptor probe, for selective sensing of Hg^{2+} . The MPDQ@poly(VPy-co-TM) sensor was attributed to the high surface area and uniform macro- and mesoporous openings of the polymer monolith. The sensor also displays color transfiguration from pale yellow to deep brown within a fast response time of ≤ 40 s, and a superior lower LOD of 0.19 ppb [51]. Likewise, Sivaraman et al. developed a two-in-one solid-state sensor for ultra-trace detection of Cu^{2+} and Co^{2+} . The dual porous bimodal poly(BTM-co-EGD) monolithic network shows uniform mesoporous tracts dispersed onto the macropore framework, facilitating uniform distribution of BTHP probe molecules. The poly(BTM-co-EGD)BTHP sensor material shows a visible color transition from pale yellow to intense olive brown for (BTHP-Cu^{2+}) and dark reddish brown (BTHP-Co^{2+}), with a detection limit of 0.13 and 0.16 ppb, for Cu^{2+} and Co^{2+} , respectively [52]. Similarly, Sivaraman et al. developed a solid-state sensor targeting Pb^{2+} . The solid-state sensor was fabricated using a porous polymer monolith template with a floral-cluster-like surface arrangement and an interwoven macro-/mesoporous network, thereby enhancing the immobilization of NDBP probe molecules. The sensor material exhibits a selective and sensitive visible color transition from pale peach to deep maroon for poly(LMC-co-EGDMA)- Pb^{2+} . It also exhibits a lower LOD of 0.28 ppb, respectively [53].

Krishna Kumar et al. synthesized a portable solid-state sensor for the selective colorimetric detection of Hg^{2+} . The poly(AA-co-TMPTA) template exhibits a ring-like porous architecture with associated multi-pore channels for the uniform encapsulation of DPQD probe molecules. The poly(AA-co-TMPTA)DPQD sensor exhibits a visible color change from light yellow to deep red upon interaction with Hg^{2+} , showing a linear response up to 100 ppb, with an

LOD of 0.23 ppb [54]. Based on a similar concept, Krishna Kumar et al. also fabricated a solid-phase optical sensor for quantifying Pb^{2+} . The sensor was fabricated using a tailor-made porous organic polymer monolith scaffold, namely poly(BM-co-PETMA), which was uniformly decorated with a chromogenic receptor (BTHB). The poly(BM-co-PETMA)BTHB sensor exhibits a color transition from pale peach to purple upon complexation with $[\text{BTHB-Pb}^{2+}]$, with an LOD of 0.31 ppb [55].

Silica and organic polymer monolith

Inorganic silica monoliths offer ordered mesoporosity and structural rigidity, enabling uniform probe immobilization. Conversely, organic porous polymer monoliths provide enhanced chemical stability and adjustable surface functionality. The use of both materials as probe-anchoring templates permits a direct comparison of inorganic and organic matrices under identical sensing conditions. The various chromoionophores immobilized on the silica and organic polymer support are depicted in Fig. 6.

With this line, Sompalli et al. synthesized three mesoporous silica monoliths (MSMs) using different SDAs, such as P123, F127, and F108. Among them, the P123 surfactant exhibited the best structural features, with a surface area of $805.68 \text{ m}^2/\text{g}$, uniform mesopores ($\sim 3.3 \text{ nm}$), and a well-defined 2D hexagonal honeycomb architecture. The continuous monolithic framework with cylindrical mesoporous channels provided enhanced mechanical stability and high accessibility for immobilizing the BBPC probe. Likewise, a polymer monolith template was synthesized, exhibiting uniform surface voids that enable efficient anchoring of the BBPC probe molecule. Both the BBPC-loaded silica and polymer monoliths facilitated rapid diffusion of Cr^{6+} , with limit of detection (LOD) values of 1.87 and 0.41 ppb, for silica and polymer sensors, respectively. It also showed a visible, naked-eye color transition from light orange to dark pink-violet [56]. Similarly, Madhesan et al. developed a solid-state opto-chemosensor for the selective detection of Hg^{2+} , with a colorimetric transition from light orange to dark red, employing MSM and MPM as templates. For this study, different MSMs were prepared via a direct template methodology using three SDAs (P123, F108, and F127) and different porogenic solvents (TiPB, TMB, *n*-decane, and *n*-dodecane). Among all the MSMs, only three combinations exhibited relatively high surface area, such as MSM-P123-TiPB ($732 \text{ m}^2/\text{g}$), MSM-F108-*n*-dodecane ($568 \text{ m}^2/\text{g}$), and MSM-F127-TiPB ($199 \text{ m}^2/\text{g}$). Of these, MSM-P123-TiPB showed the highest surface

area, with a pore volume of $0.8 \text{ cm}^3/\text{g}$ and a pore size of 2.81 nm. In addition, HR-TEM analysis of MSM-P123-TiPB revealed a hierarchical macroporous framework interconnected by mesopores. The mesoporous polymer monolith was also used as a template, exhibiting a uniformly distributed, well-ordered, interconnected porous network that facilitates the homogeneous distribution of the HBPQ probe throughout the silica and polymer monoliths. The resulting HBPQ-MSM and HBPQ-MPM sensors demonstrated efficient colorimetric detection of Hg^{2+} within ≤ 2 min of contact time, with LODs of 0.2 & 0.1 ppb [57].

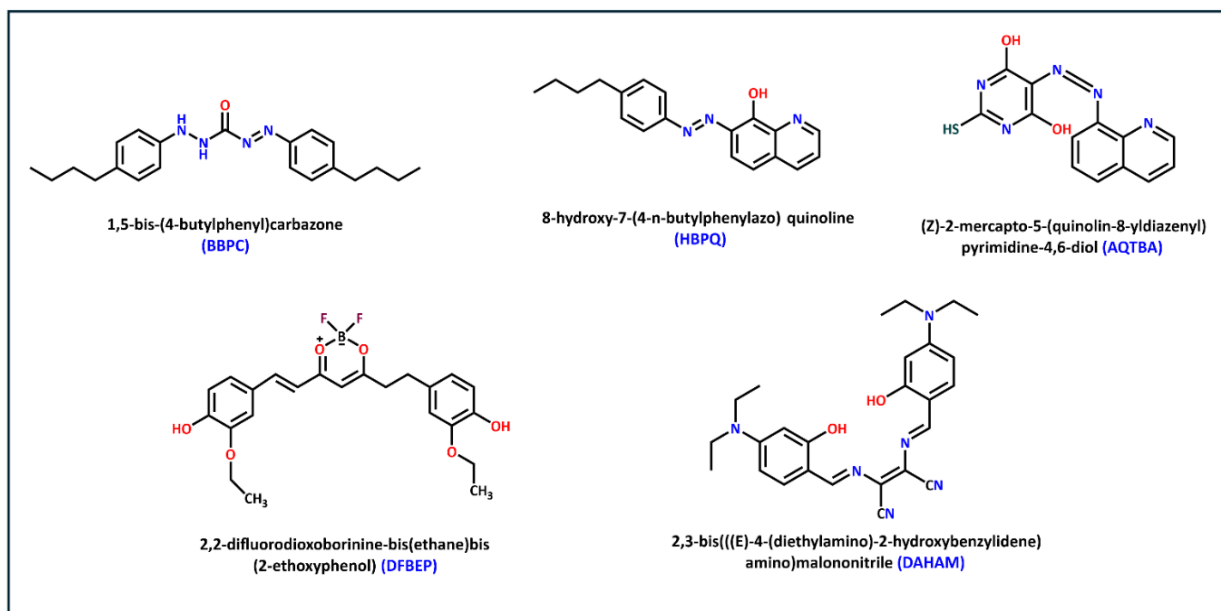


Fig. 6. List of chromoionophoric probes for immobilization onto a silica and organic monolith.

In addition, Kongasseri et al. developed a silica and polymer-based sensor system for detecting Co^{2+} . Both templates facilitate the homogeneous distribution of AQTBA probe molecules. The opto-chemosensor exhibited maximum ion-sensing performance at pH 7.0, with a color change from pale yellow (blank) to reddish brown upon complexation with Co^{2+} , and LODs of 0.35 & 0.07 ppb for the silica & polymer sensors [58]. Sompalli et al. fabricated a continuous, highly ordered mesoporous silica monolith (MSM) using a direct templating approach with TEOS and Pluronic F108, in which controlled addition of n-dodecane enabled the formation of a stable microemulsion. The resulting MSM exhibited superior structural and surface properties, enabling efficient immobilization of the DFBEP ligand within the

systematically arranged, interconnected cubic porous network of the 2D silica monolith, thereby yielding a silica-based sensor material (Optode-I). Likewise, Optode-II was prepared using a porous organic polymer monolith as a template, which exhibits a fused porous framework, comprising interconnected meso-tracts within a uniformly distributed macroporous framework immobilizing DFBEP probe molecules. As shown in Fig. 7, both Optode I and II exhibit ultra-trace detection of As^{3+} , accompanied by a distinct naked-eye color transition from pale pink to deep purple. Furthermore, the sensors exhibited a linear response range of 2-300 ppb with a LOD of 0.87 and 0.75 ppb [59].

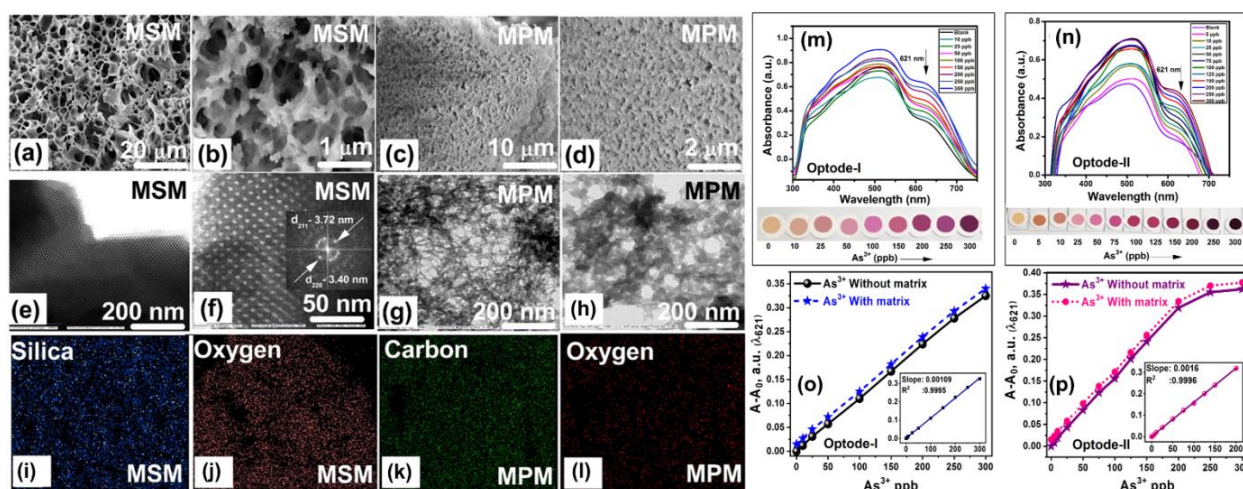


Fig. 7. (a-h) FE-SEM and HR-TEM images of the MSM and MPM templates; (i-l) Elemental mapping of the MSM and MPM templates; (m&n) UV-Vis-DRS signal response and color change; and (o&p) calibration plot and linear fit for various As^{3+} concentrations for Optode-I and Optode-II.

Kongasseri et al. prepared a silica and polymer monolithic framework (MF) as a template for a sensor system, which was anchored with DAHAM probe molecules for sensing of ultra-trace concentration of Cu^{2+} . The SMF exhibits a uniform surface morphology, voluminous porosity, and excellent stability features. The surface morphology exhibits a fibril-like porous pattern and a continuous, interconnected honeycomb-like arrangement of mesopores. Conversely, the PMF exhibits deep, intertwined cave-like patterns. Both the SMF and PMF facilitate the diffusion of Cu^{2+} to the uniformly distributed DAHAM probe molecules. The SMF and PMF sensors exhibit a visible color transition from deep orange to dark green, with an LOD of 0.75 and 0.11 ppb, respectively [60].

Chromoionophore functionalized polymer monolith and silica-embedded solid-state sensor

Sompalli et al. demonstrated a dual-template solid-state colorimetric sensing strategy for Co^{2+} ions by employing two structurally distinct porous hosts: mesoporous silica monoliths and macro- and mesoporous polymer monoliths functionalized with different chromoionophoric ligands. The silica monolith was impregnated with BDHCA, in which Co^{2+} recognition occurred via physical adsorption and ligand-to-metal charge-transfer interactions, resulting in a color change from pale yellow to greenish brown to the naked eye. In contrast, the polymer monolith incorporated ATDBD as a functional monomer covalently embedded within the network, yielding a pale orange to deep red response with superior sensitivity. Optimization of ion-sensing performance yields detection limits of 0.29 and 0.15 ppb for Co^{2+} using silica- and polymer-based monolithic sensors, respectively [61]. The chromoionophores immobilized onto the monolith are depicted in Fig. 8.

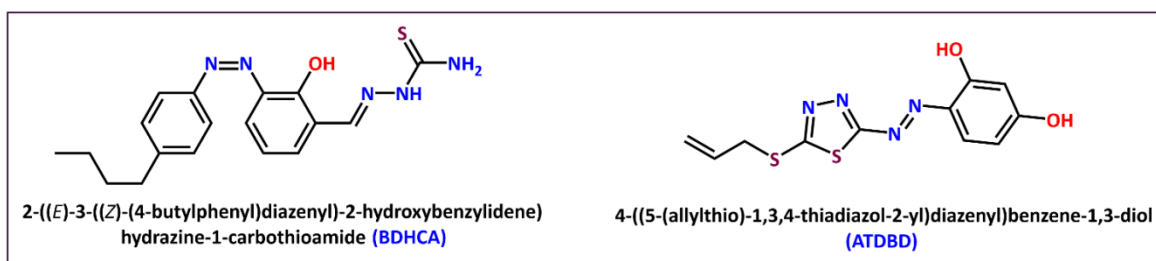


Fig. 8. List of chromoionophoric probes used for immobilizing and functionalizing as monoliths.

Hybrid monoliths

Sangeetha et al. first demonstrated the potential of hybrid organic-inorganic polymer monoliths for solid-state colorimetric sensing by immobilizing the chromoionophoric ligand HQDB for selective detection of Cu^{2+} ions. The sensor exhibited a distinct color change from yellowish-orange to dark brown, arising from the formation of a stable 1:1 $[\text{Cu}^{2+}\text{-HQDB}]$ charge-transfer complex, with an absorption maximum at 480 nm. The porous hybrid framework promoted efficient probe-metal interactions, enabling sensitive detection with a reported limit of detection of 0.45 ppb [62]. Building on this platform, the same author extended the hybrid monolith approach to multi-ion sensing by incorporating the Azo-based ligand TAN, thereby enabling visual discrimination among Pb^{2+} , Hg^{2+} , and Cd^{2+} ions. Upon metal binding, the monolith exhibited color transitions from light orange to brown for Pb^{2+} , reddish maroon for Hg^{2+} , and purplish pink for Cd^{2+} , with LODs of 0.42, 0.50 and 0.48 ppb, respectively [63]. Anju et al.

advanced earlier hybrid monolithic colorimetric sensing approaches by employing a hydrolyzed organic-inorganic hybrid monolith for selective UO_2^{2+} detection. Controlled hydrolysis generated surface -Si-OH groups within the poly(TMSPMA-co-EGDMA) framework, enhancing receptor anchoring and accessibility. A spirocyclic azomethine receptor (DHDTBX) was immobilized in a porous matrix, resulting in a distinct color transition from ivory to dark beige with linearity over 1.0-150 $\mu\text{g/L}$ and LOD of 0.15 ppb. The hydrolyzed monolith was reusable for 9 cycles with $\text{RSD} \leq 2.1\%$, confirming robustness [64]. The chromoionophores immobilized onto the hybrid monolith are depicted in Fig. 9.

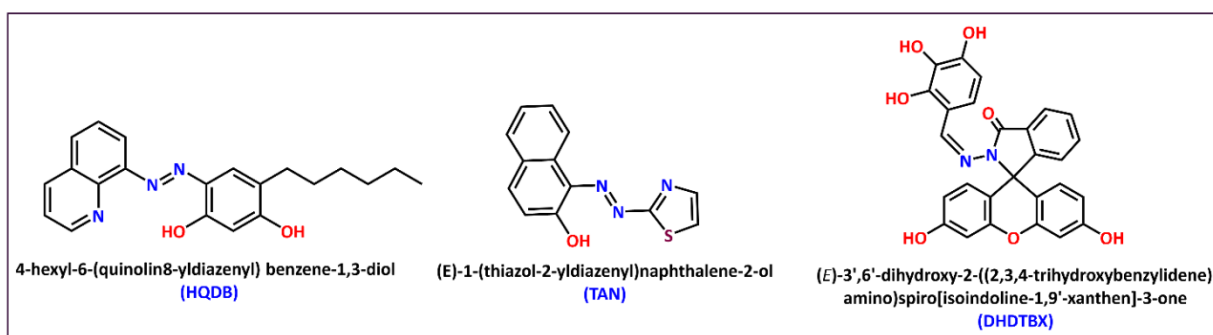


Fig. 9. List of chromoionophoric probes for immobilization onto a hybrid monolith.

Metal Organic Frameworks (MOFs) and Polymer Monolith

Kuppusamy et al. reported a solid-state colorimetric sensing platform based on an Al-based metalorganic framework functionalized with Punica granatum peel extract (PPE) for selective detection of Cr(VI). The PPE-loaded MOF exhibited a clear color transition from light yellow to dark brown due to polyphenol-mediated reduction of Cr(VI) to Cr(III), followed by coordination within the MOF pores. The sensor showed linear detection over 0.3-200 ppb with a LOD of 0.23 ppb, along with good reusability and low RSD. In contrast to earlier monolithic systems employing synthetic chromoionophoric ligands, this work uniquely demonstrates the use of a naturally derived extract as a green sensing component, effectively harnessing MOF porosity and confinement effects for sustainable solid-state colorimetric sensing [65].

Kuppusamy et al. demonstrated a dual-template solid-state colorimetric sensing strategy by immobilizing the same chromoionophoric ligand, DPPE (Fig. 10), onto two distinct porous hosts: an Al-based MOF (MIL-53(Al)) and a porous polymer monolith, thereby enabling comparative detection of Cr^{3+} . Both platforms exhibited a concentration-dependent color

transition from pale orange to sepia-brown. Notably, the MOF sensor showed a slightly higher LOD (0.53 ppb) than the polymer monolith (0.44 ppb), reflecting the influence of pore architecture and surface area on sensing performance. At the same time, both systems retained reusability up to eight cycles with low RSD [66].

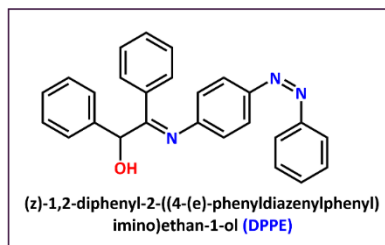


Fig. 10. The chromoionophoric probe used for immobilizing onto a MOF and polymer-based template.

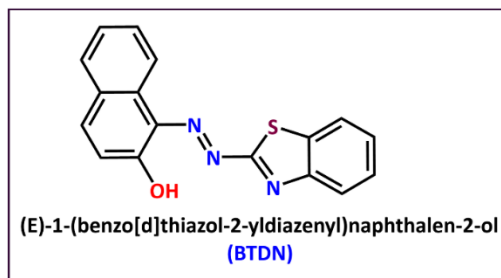


Fig. 11. The chromoionophoric probe for immobilizing onto a hybrid monolith and MOF-based template.

MOF and hybrid monolith

Anju et al. reported a comparative solid-state colorimetric sensing strategy in which the chromoionophoric ligand BTDN (Fig. 11) was immobilized on two structurally distinct porous hosts: microporous UiO-66 MOF and a meso/macroporous hydrolyzed hybrid poly(TMSPMA-co-TMPTA) monolith, for ultra-trace Pb^{2+} detection. Both platforms exhibited a concentration-dependent color transition from apricot to vivid claret, driven by the formation of the Pb^{2+} -BTDN charge-transfer complex as shown in Fig. 12. The BTDN@poly(TMSPMA-co-TMPTA) and BTDN@UiO-66 sensors exhibited linear ranges of 0.1-200 ppb with LODs of 0.14 and 0.27 ppb, respectively, with excellent reusability for up to 16 cycles, demonstrating that pore hierarchy and surface chemistry influence sensing performance [67].

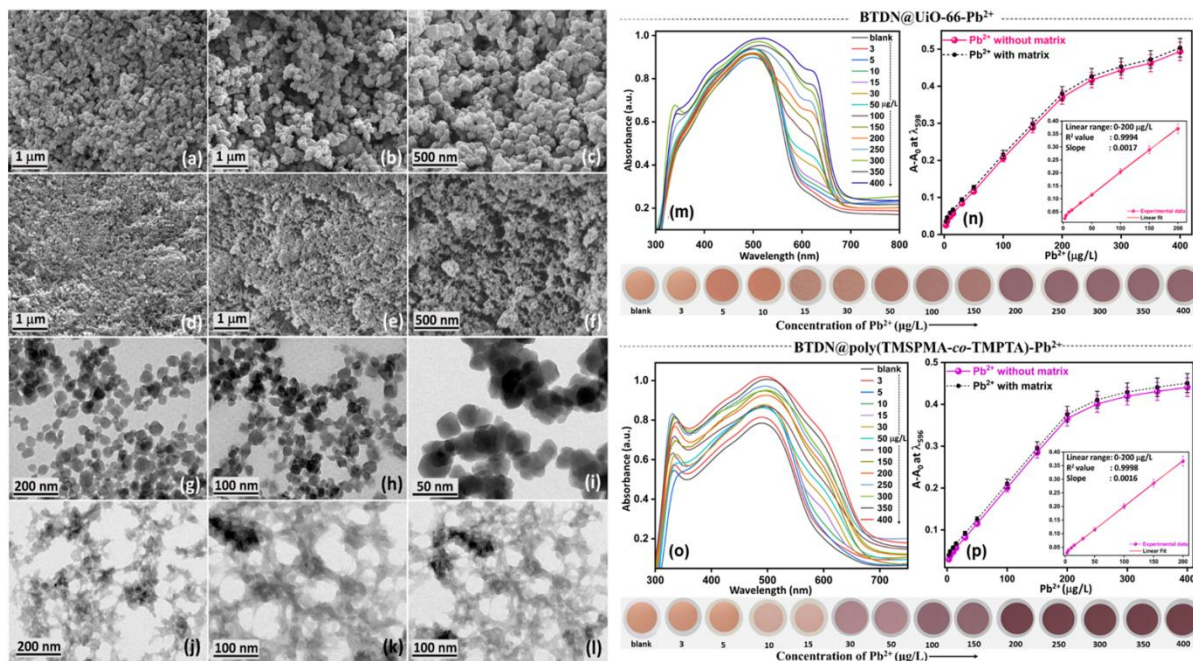


Fig. 12. FE-SEM images of **(a-c)** UiO-66 and **(d-f)** poly(TMSPMA-co-TMPTA), HR-TEM images of **(g-i)** UiO-66 and **(j-l)** poly(TMSPMA-co-TMPTA), **(m-p)** UV-Vis-DRS signal response and calibration plot for BTDN@UiO-66-Pb²⁺ and BTDN@poly(TMSPMA-co-TMPTA)-Pb²⁺.

Conclusions

Solid-state colorimetric sensors have evolved into reliable and versatile platforms for heavy-metal ion detection, combining visual simplicity with material-driven performance enhancements. This review highlights how monolithic and porous templates critically influence sensing efficiency through controlled pore architecture, surface chemistry, and probe immobilization. Silica monoliths provide ordered porosity and rapid response, while organic polymer monoliths offer superior mechanical stability and chemical robustness, enabling repeated use. Hybrid and hydrolyzed hybrid monoliths further enhance sensing performance by integrating organic flexibility with inorganic rigidity, with surface hydroxylation enhancing probe anchoring, sensitivity, and durability. Metal-organic frameworks expand these capabilities through high surface areas, tunable pores, and confinement effects that amplify optical responses. Comparative dual-template strategies using identical ligands across different platforms reveal clear relationships between structural performance and sensor design, guiding rational sensor design. Collectively, these developments demonstrate that solid-state colorimetric sensors are approaching practical readiness. Future research is expected to further strengthen this

field through sustainable probe design, scalable material synthesis, and integration with portable readout technologies. With continued innovation, monolithic and porous solid-state colorimetric sensors are poised to become reliable, user-friendly solutions for environmental monitoring and heavy metal ion detection.

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Chemical Separation Strategies in Nuclear Medicine: From Radionuclide Production to Radiopharmaceutical Formulation

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Introduction

Radiopharmaceuticals are a specialized class of radiochemical formulations with defined and consistent composition, suitable for safe administration to humans - either orally or intravenously - for the diagnosis or treatment of disease [1]. They play a crucial role in nuclear medicine by enabling non-invasive visualization of physiological and biochemical processes at the molecular level, as well as targeted treatment of various diseases, predominantly cancer. A radiopharmaceutical typically consists of two main components: a radionuclide that emits radiation suitable for imaging or therapy, and a carrier molecule (ligand) that directs the radionuclide to a specific biological target [2,3]. Figure-1 depicts the basic structure of a typical radiopharmaceutical.

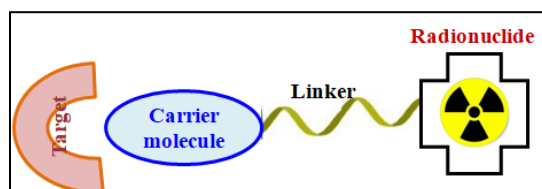


Figure 1. Basic structure of a typical radiopharmaceutical

Diagnostic radiopharmaceuticals generally employ either gamma emitting radionuclides (Tc-99m, In-111, I-123 etc.) or positron emitting radionuclides (Ga-68, F-18, Zr-89 etc.) [4-6]. Gamma radiations are characterized by low linear energy transfer (LET), enabling them to pass through body tissues and be detected by radiation detectors positioned outside the patient. For positron-emitting radionuclides, the emitted positron combines with an electron in the surrounding medium, resulting in an annihilation event that produces two 511 keV gamma photons emitted in opposite directions and detected in coincidence for image reconstruction.

Depending on the nature of the emitted radiation, nuclear medicine imaging is carried out using either single-photon emission computed tomography (SPECT) or positron emission tomography (PET).

Therapeutic radiopharmaceuticals, on the other hand, utilize particulate (beta, alpha or Auger electron) emitting radionuclides such as, Lu-177, Y-90, I-131, Ac-225 to deliver cytotoxic doses of ionizing radiation to the diseased site. In therapeutic radiopharmaceuticals, particulate emitting radionuclide is tagged to monoclonal antibodies or peptides or small molecules to achieve targeted delivery of cytotoxic ionizing radiation [7-10].

In both diagnostic and therapeutic applications, the chemical purity, radionuclidic purity and radiochemical purity, as well as biological specificity of a radiopharmaceutical are key determinants of its safety and efficacy. Consequently, separation chemistry plays a central role at every stage of radiopharmaceutical development and formulation, starting from radioisotope production to ligand synthesis, radiolabeling, and final product purification, ensuring that the desired radioactive species is obtained in a chemically and biologically acceptable form, suitable for human administration.

Role of separation science in radioisotope production

Radioisotopes used in nuclear medicine are mainly produced in nuclear reactors or cyclotrons by neutron activation, nuclear fission, or charged particle induced reactions. Following irradiation, the target undergoes radiochemical processing which may employ a range of separation techniques to isolate the desired radioisotope from unreacted target material, byproducts, and other impurities. The presence of radiochemical and radionuclidic impurities can hinder radiopharmaceutical preparation by lowering radiolabeling yields and generating undesired products, which may increase the radiation dose burden to the patient and negatively impact scintigraphic image quality [11]. Separation chemistry is fundamental to the isolation of the desired radionuclide in a form that meets stringent requirements for radionuclidic, radiochemical, and chemical purity, thereby ensuring its suitability for subsequent radiopharmaceutical preparation. For example, Mo-99 produced via the fission of U-235 must be separated from numerous co-produced fission products and residual uranium. Many of these fission fragments are highly radioactive, and some are volatile, which makes the radiochemical isolation of Mo-99 particularly challenging. To obtain clinical-grade Mo-99 from irradiated U-235 targets, a

combination of separation techniques, including target dissolution, filtration, ion-exchange chromatography, and solvent extraction, is employed. I-131 produced through neutron irradiation of Te-130 must be separated from unreacted tellurium and other byproducts, which is done by exploiting the volatility of iodine. The irradiated Te-130 target is heated to release iodine through distillation, which is subsequently trapped in a suitable solution, leaving tellurium behind. Additional purification steps ensure that the final product meets the required radionuclidic and chemical purity for clinical applications.

Separation chemistry plays a crucial role in the production of Lu-177 from irradiated Yb targets by enabling the efficient and selective isolation of the desired radioisotope with high purity. After neutron irradiation of enriched Yb-176, the target contains a complex mixture of bulk Yb, newly formed Lu-177, and trace radioactive and stable impurities. Because Lu and Yb are chemically similar lanthanides, advanced separation techniques, most commonly ion-exchange chromatography or extraction chromatography, are required to exploit subtle differences in their ionic behavior. Effective separation chemistry ensures high radionuclidic and chemical purity of Lu-177, minimizes Yb breakthrough, and allows recovery and recycling of the expensive enriched Yb target material. This step is therefore essential not only for meeting the stringent quality requirements for medical applications of Lu-177, but also for improving process efficiency, cost-effectiveness, and sustainability of large-scale radioisotope production.

Radionuclide generators are an important source of radioisotopes for nuclear medicine, providing on-site access to short-lived clinically useful radionuclides derived from long-lived parent isotopes. These generators operate on the principle of radioactive equilibrium, allowing the daughter radionuclide to be repeatedly separated at regular intervals by exploiting the chemical differences between parent and daughter radionuclides. Various separation techniques, such as, column chromatography, solvent extraction, distillation, membrane filtration, and electrochemical methods are employed to isolate the daughter radionuclide from the parent. Separation science is critical in this process, as it enables selective isolation of the short-lived daughter, minimizes parent breakthrough, and reduces other radioactive or chemical impurities, thereby ensuring high radionuclidic and chemical purity. Consequently, effective separation is essential for the reliable operation of radionuclide generators and their safe, efficient use in clinical nuclear medicine.

Role of separation science in the synthesis of carrier moieties

The synthesis of carrier moieties is a crucial step in the development of radiopharmaceuticals, as the carrier largely governs the biological targeting and pharmacokinetic behavior of the final radiolabeled product. Carrier moieties may include small molecules, peptides, proteins, antibodies, or nanoparticles, and are commonly functionalized with appropriate chelating agents to allow efficient and stable radiolabeling with various radionuclides. Separation science is a vital component in the synthesis of carrier moieties, as it ensures the efficient isolation, purification, and characterization of the desired carrier systems. During synthesis, separation techniques are employed to remove unreacted materials, by-products, and impurities, thereby improving the purity and quality of the carrier moiety.

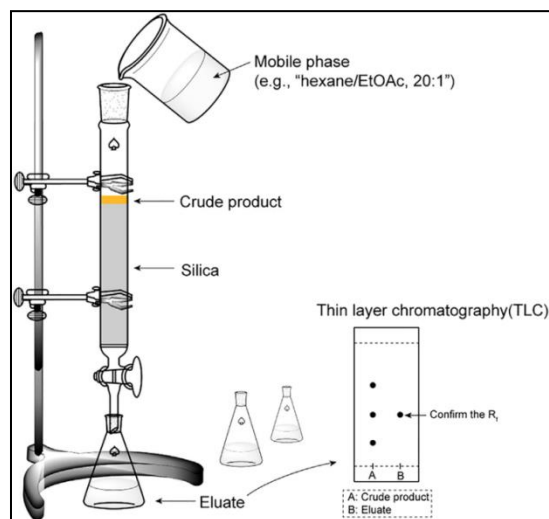


Figure-2. Process of column chromatography technique

Separation chemistry is integral to every stage of ligand synthesis, from monitoring the progress of the reaction to the purification of the final product. In small-molecule synthesis, thin-layer chromatography (TLC) is routinely used at each reaction stage to monitor product formation and the consumption of starting materials. Differences in polarity cause reactants and products to interact differently with the stationary and mobile phases, leading to their separation. In TLC, a small amount of the sample is applied to a plate coated with silica gel or aluminium oxide and developed in an appropriate solvent system. As the solvent moves upward, the components migrate at different rates depending on their relative interactions with the stationary phase and the solvent. By comparing the positions of the spots corresponding to the starting

materials and the product, along with their retention factor (R_f) values, reaction progress and the presence of impurities can be readily assessed. The R_f value of the desired product is subsequently used to guide its quantitative separation from unreacted materials and by-products through preparative TLC or column chromatography employing silica gel or aluminium oxide as the stationary phase. Figure-2 shows the process of column chromatography technique used for purification in case of small molecule synthesis.

Peptide-based ligands, including somatostatin analogs such as DOTA-TATE and DOTA-TOC, as well as prostate-specific membrane antigen (PSMA) targeting peptides, are commonly synthesized using solid-phase peptide synthesis. After synthesis, the peptide is cleaved from the solid resin, producing a crude mixture. This crude product is subsequently purified by reverse-phase high-performance liquid chromatography (RP-HPLC) to remove truncated sequences, side products, and residual protecting groups. In RP-HPLC, separation is achieved using a non-polar stationary phase, typically a C_8 or C_{18} (octyl- or octadecylsilane) column, in combination with a polar mobile phase composed of water and an organic solvent such as, acetonitrile or methanol. Upon injection of the crude peptide mixture into the HPLC system, individual components interact differently with the non-polar stationary phase according to their hydrophobicity. Consequently, they elute at distinct retention times, allowing efficient separation of the desired peptide from impurities. The retention times of the analytes are monitored by detecting their UV-visible absorption at a wavelength characteristic of the target peptide. Thus, separation science plays a critical role in ensuring the purity, identity, and suitability of peptide-based carrier moieties for their subsequent use in radiopharmaceutical development. A schematic representation of a RP-HPLC system coupled with a UV-Vis detector is shown in Figure 3.

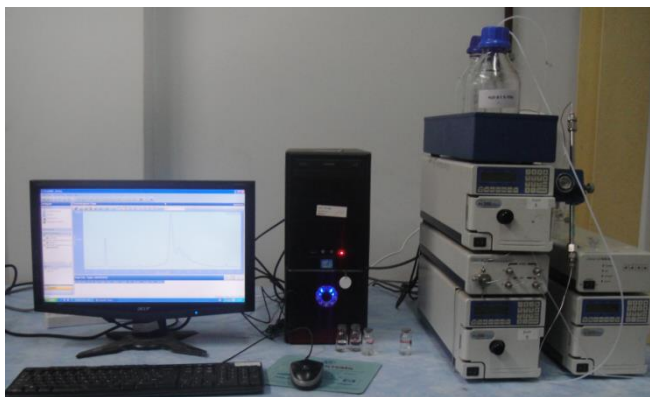


Figure-3. RP-HPLC system coupled with UV-Vis detector

For high molecular weight species such as antibodies, antibody fragments, and nanoformulations, size-exclusion chromatography (SEC) is commonly employed for purification. SEC separates molecules on the basis of their molecular size using a column packed with porous beads of a defined pore size. Larger molecules, which are unable to enter the pores, elute first, whereas smaller molecules penetrate the pores, follow a longer path through the column, and elute at later times.

In addition to above-mentioned methods, several other separation techniques are utilized during the purification of carrier moieties. These include Distillation, which exploits differences in boiling points of components in a liquid mixture; Recrystallization, which is based on temperature-dependent solubility differences; Extraction, which separates compounds according to their relative solubilities in two immiscible solvents; Sublimation, where a solid directly transitions to the gas phase without passing through a liquid state; Filtration, which is used to remove solid impurities from liquids or gases using a porous medium; and Drying, which is employed for removal of residual solvents, particularly water, using suitable drying agents such as, anhydrous Na_2SO_4 , MgSO_4 , or CaCl_2 .

The chemical purity of the ligand critically influences radiolabeling efficiency, radiochemical yield, and *in-vivo* stability. The presence of impurities may compete with the ligand for radionuclide coordination or adversely affect biodistribution. Hence, the implementation of robust and reproducible separation techniques during ligand synthesis is essential to obtain highly pure ligands suitable for radiopharmaceutical formulation and safe clinical application.

Role of separation science in formulation of radiopharmaceuticals

Radiopharmaceuticals are typically prepared by incorporating the desired radionuclide into a specific carrier moiety through a process known as radiolabeling. This step must be rapid, efficient, and highly reproducible. Separation science plays a crucial role in purifying the radiolabeled product to ensure its suitability for clinical use. Following radiolabeling, the reaction mixture may contain free radionuclide, hydrolyzed species, unlabeled ligand, and radiolytic degradation products. To assess and ensure radiochemical purity, chromatographic techniques such as thin layer chromatography (TLC), paper chromatography (PC), and radio-high-performance liquid chromatography (radio-HPLC) are routinely employed.

In radio-HPLC, a radioactive detector, typically a NaI(Tl) detector, is integrated with the HPLC system to monitor radiolabeled species. After radiolabeling, a small aliquot of the reaction mixture (usually 10-20 μL) is injected into the system. Different radiolabeled compounds separate and elute at distinct retention times, allowing the radiochemical purity and yield to be calculated from the relative peak areas of the desired product in the radio-HPLC chromatogram. Figure-4 illustrates the radio-HPLC profile of Lu-177-labeled DOTA-TATE, where the ^{177}Lu -DOTA-TATE elutes at a retention time (R_t) of 16.8 minutes, while free $^{177}\text{LuCl}_3$ appears between 3-4 minutes.

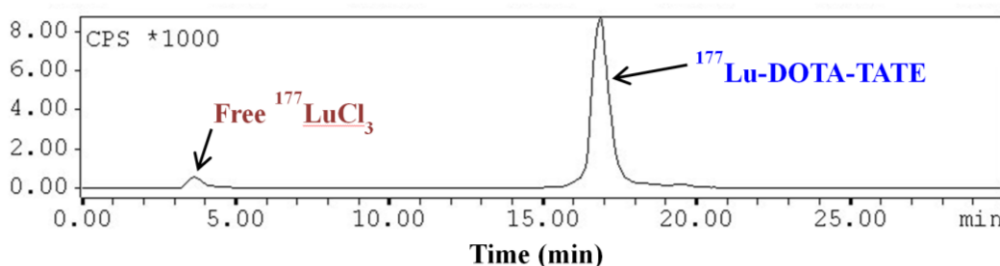


Figure-4. Radio-HPLC chromatogram of Lu-177 labeled peptide (DOTA-TATE)

In ITLC (instant thin layer chromatography) and PC, the radiolabeled product, free radionuclide, and hydrolyzed species migrate to different positions on the strip when developed in an appropriate solvent. The distribution of radioactivity is then measured using a radio-TLC scanner (a TLC scanner coupled with a NaI(Tl) detector) or a gamma counter. The radiochemical yield is calculated as the percentage of total radioactivity corresponding to the desired radiolabeled product.

Since radiopharmaceuticals are intended for administration in living subjects, their radiochemical purity should ideally be 95% or higher. If this level of purity is not achieved after radiolabeling, post-labeling purification becomes necessary. For instance, F-18 radiochemistry relies heavily on separation techniques, as efficient removal of unreacted precursors, side products, and residual reagents is required after radiolabeling to achieve high radiochemical purity and optimal molar activity.

A variety of separation techniques are employed to purify radiopharmaceuticals depending on their nature. For small-molecule and peptide-based radiopharmaceuticals, pre-packed C_{18} cartridges are commonly used to remove free or unbound radiometal. In contrast, for

radiolabeled antibodies and nanoformulations, free radiometal is typically separated using cyclodextrin-based G-25 resin, which operates on the principle of size-exclusion chromatography.

These examples highlight that separation science is fundamental to the production of radiopharmaceuticals, ensuring that the final formulations are highly pure, safe, and suitable for clinical application.

Conclusion

Separation chemistry is a cornerstone of radioisotope production and radiopharmaceutical preparation. It enables the efficient isolation of radionuclides, the synthesis and purification of targeting ligands, and the production of high-quality radiolabeled compounds suitable for clinical application. Recent advances in separation materials and automated processes are enhancing both the efficiency and safety of these procedures. As nuclear medicine continues to advance toward more personalized and targeted therapies, separation science will remain a critical driver in the development of next-generation radiopharmaceuticals, ensuring they are safe, effective, and readily accessible for patient care.

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Cloud Point Extraction for Separation and Preconcentration of Metal Ions: Applications in Environmental and Nuclear Chemistry

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Introduction

Over the past two decades, cloud point extraction (CPE), also referred to as micelle-mediated extraction (MME), has emerged as a simple, cost-effective, and environmentally benign technique for the separation and pre-concentration of metal ions from complex matrices, particularly environmental samples [1–5]. The technique was first introduced in 1976 by Watanabe and co-workers, who demonstrated its applicability for the pre-concentration of metal ions through the formation of hydrophobic metal–ligand complexes [6]. Subsequently, CPE gained wide acceptance in biochemical and analytical applications, including the purification and separation of proteins and biomolecules [4].

CPE is based on the phase separation behavior of aqueous surfactant solutions. Certain non-ionic and zwitterionic surfactants form clear and homogeneous micellar solutions at low temperatures; however, upon reaching a characteristic temperature known as the cloud point temperature (CPT), the solution becomes turbid and separates into two isotropic, immiscible phases [1–5]. These phases consist of (i) a dilute aqueous phase containing surfactant at a concentration close to or below the critical micelle concentration (CMC), and (ii) a surfactant-rich phase (SRP) of comparatively small volume. Although temperature is the most commonly employed trigger for phase separation, similar behavior can also be induced by changes in pressure or by the addition of electrolytes and salting-out agents [1–5].

Analytes that exhibit affinity towards the micellar core, either through inherent hydrophobicity or via conversion into hydrophobic complexes, preferentially partition into the SRP. In the case of metal ions, selective complexation with suitable ligands transforms hydrated metal species into neutral or weakly charged hydrophobic complexes, thereby enabling efficient separation and pre-concentration using CPE. Owing to the small volume of the SRP, significant enrichment factors can be achieved without the use of large volumes of organic solvents. CPE has gained recognition as an eco-friendly pre-concentration and separation technique for the

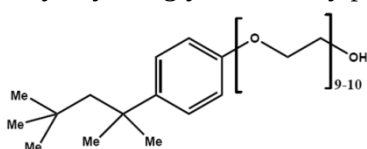
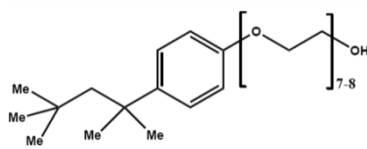
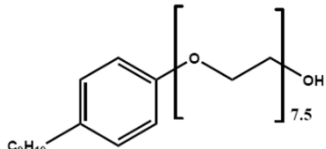
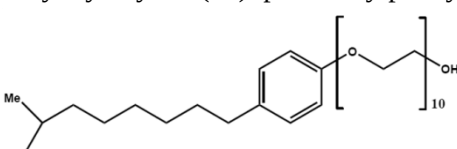
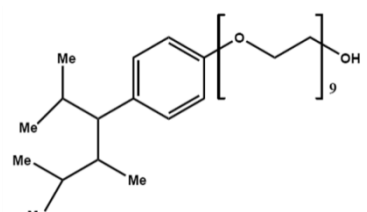
detection and remediation of heavy metal ions, largely due to its compliance with the principles of green chemistry [1,5]. The method has also found applications in nuclear research, particularly in the trace impurity characterization of nuclear fuels and related materials. One of the primary challenges in such applications is the reduced stability of micelles under highly acidic conditions typical of nuclear fuel dissolution media. This limitation has been addressed through the use of surfactant mixtures, micelle surface modifiers, and specially designed ligands capable of stabilizing the micellar system in harsh chemical environments. In addition, CPE shows promise for the stripping and recovery of uranium species from liquid organic waste streams, offering a greener alternative to conventional liquid–liquid extraction (LLE). Unlike LLE, which relies on large volumes of volatile and often toxic organic solvents, CPE employs small quantities of relatively non-toxic, non-volatile, and non-flammable surfactants, resulting in minimal secondary waste generation and enhanced operational safety [1,5]. The successful application of CPE to diverse sample matrices depends on several experimental parameters, which are discussed in the following sections.

CPE: Role of non-ionic surfactant

Surfactants are amphiphilic molecules composed of a hydrophilic head group and a hydrophobic tail, and are broadly classified into four categories: non-ionic, cationic, anionic, and zwitterionic types [1–5,7]. The hydrophobic tail generally consists of long-chain hydrocarbons, branched alkyl groups, or hydrocarbon chains containing aromatic rings, whereas the hydrophilic head group may be ionic or polar in nature. In aqueous media, when surfactants are present at concentrations above their critical micelle concentration (CMC), the individual surfactant monomers self-assemble into organized aggregates known as micelles [1–5,7]. This self-assembly is driven primarily by the minimization of unfavorable interactions between the hydrophobic tails and water molecules. Structurally, a micelle consists of two distinct regions: a hydrophobic core and a hydrophilic corona. The micellar core provides a non-polar microenvironment capable of solubilizing hydrophobic species, including neutral or weakly charged metal–ligand complexes, and this property forms the basis of cloud point extraction. The hydrophilic head groups constitute the micellar corona and remain in direct contact with the surrounding aqueous phase through hydrogen bonding and dipole–dipole interactions, forming a solvation shell around the micelle. Upon heating the system to the cloud point temperature

(CPT), dehydration of the hydrophilic head groups occurs due to the disruption of these hydrogen bonds. As a consequence, micelles lose their hydration layer, aggregate, and separate from the bulk aqueous phase to form a surfactant-rich coacervate phase.

Table 1. List of commonly used nonionic surfactants along with their CMC and CPT

Surfactant	Commercial name	CMC (mmol L ⁻¹)	CPT (°C)
	Triton X-100 (TTX-100)	0.17-0.30	64-65
	Triton X-114 (TTX-114)	0.20-0.35	23-25
	PONPE-7.5	0.085	5-20
	PONPE-10	0.07-0.085	62-65
	IGEPAL CO-630	---	48-52

Non-ionic and zwitterionic surfactants are most commonly employed in CPE procedures [1–5]. In contrast, cationic and anionic surfactants are generally unsuitable for cloud point extraction because strong electrostatic repulsion between similarly charged head groups hinders effective micellar aggregation and phase separation. Non-ionic surfactants, in particular, exhibit

well-defined cloud point behavior and relatively low sensitivity to ionic strength, making them highly attractive for analytical and separation applications.

A variety of surfactants have been employed by different research groups for CPE, and their key physicochemical properties, such as CMC and CPT, are summarized in Table 1. Knowledge of these parameters is essential for selecting an appropriate surfactant, or a suitable surfactant mixture, when developing new CPE methodologies. The majority of surfactants reported for the cloud point extraction of metal ions are non-ionic in nature, typically comprising hydrophilic polyoxyethylene chains coupled with hydrophobic alkyl or aromatic groups, which together impart favorable micellization and phase separation characteristics.

CPE: Role of metal coordinating ligands

Cloud point extraction of metal ions generally requires the presence of an appropriate chelating agent capable of converting hydrated metal ions into extractable metal chelates [1–5]. Since hydration strongly stabilizes metal ions in aqueous media, ligands with sufficient hydrophobic character are preferred over highly hydrophilic chelating agents, as they promote effective partitioning of the resulting metal complexes into the surfactant-rich phase.

In a micellar medium, a hydrophobic chelating agent is readily solubilized within the micellar core. Owing to its amphiphilic nature, the hydrophobic moiety of the ligand remains embedded in the core, while the metal-coordinating functional group is oriented toward the micellar corona, where it remains accessible to metal ions present in the aqueous phase [8]. This spatial arrangement facilitates efficient metal–ligand interactions at the micelle–water interface and eliminates the need for vigorous mixing or external phase-contacting forces that are typically required in conventional liquid–liquid extraction (LLE). A schematic representation illustrating the orientation of ligand molecules within the micellar structure, as well as the overall cloud point phase separation process, is shown in Figure 1. Favre-Réguillon et al. demonstrated that, under identical acidic conditions, cloud point extraction of uranium using 8-hydroxyquinoline exhibits significantly higher extraction efficiency than liquid–liquid extraction employing the same ligand [9]. This enhancement is attributed to the favorable microenvironment provided by the micellar system, which promotes complex formation and efficient phase transfer.

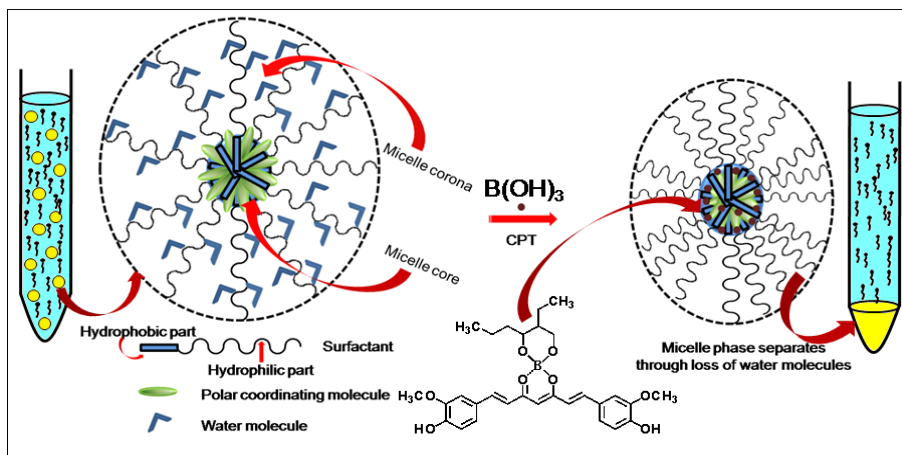


Fig.1. Schematic representation of the CPE procedure

The hydrophobic interior of the micellar core is particularly suited for the extraction of neutral metal chelates. However, several studies have reported successful extraction of charged metal complexes through modification of micellar systems, such as the use of mixed surfactants, ion-pairing agents, or micelle surface modifiers [10–12]. These strategies expand the applicability of CPE to a wider range of metal speciation conditions. Consequently, the selection of metal-coordinating ligand(s) is a critical factor in the design of CPE methodologies, as it directly influences extraction efficiency, selectivity, and compatibility with specific chemical matrices. Careful ligand design and optimization are therefore essential, particularly for complex systems such as actinide and nuclear fuel-related samples.

CPE: Role of phase modifiers

Modification of the micellar system is often necessary to enable the extraction of charged metal chelates into the hydrophobic micellar core. This is commonly achieved through the addition of ionic co-surfactants or, less frequently, ionic liquids (ILs) [10–12]. In addition to facilitating extraction, such modifiers can significantly influence key physicochemical parameters of the micellar system, including the critical micelle concentration (CMC) and the cloud point temperature (CPT) of non-ionic and zwitterionic surfactants [13,14].

When an ionic co-surfactant is introduced into a micellar medium at concentrations below its own CMC, the surface-active ions may exist either as monomers in solution or become incorporated into mixed micelles formed with the primary surfactant. Among the various co-surfactants investigated, cetyltrimethylammonium bromide (CTAB) and sodium dodecyl sulfate

(SDS) are the most widely employed in CPE studies [10–12]. In the mixed micellar state, these modifiers enhance metal extraction by introducing electrostatic interactions between the micelle surface and oppositely charged metal complexes, and in some cases by directly participating in the metal coordination process.

The presence of an oppositely charged hydrophilic head group of the co-surfactant can partially or completely neutralize the charge of the metal chelate, thereby increasing its effective hydrophobicity and promoting its partitioning into the surfactant-rich phase. Labrecque et al. [10] and Madrakian et al. [11] reported quantitative extraction of U(VI) using P,P'-di(2-ethylhexyl) methanediphosphonic acid and pyrocatechol violet, respectively, in the presence of CTAB, highlighting the effectiveness of cationic co-surfactants in uranium extraction systems. In contrast, the use of ionic liquids as phase modifiers in CPE has received limited attention. To date, only a single report by Gao et al. has demonstrated the effect of ionic liquid addition on the CPE of U(VI) [12]. In this study, the incorporation of a hydrophobic ionic liquid containing bis(trifluoro-methane-sulfonyl)imide (NTf₂⁻) as the anionic component was shown to enhance uranium extraction. The improvement was attributed to the incorporation of the ionic liquid into the micellar phase and the neutralization of the charged U(VI)–trioctylphosphine oxide complex through the participation of the NTf₂⁻ anion in the overall complexation process. These studies clearly indicate that the choice of phase modifier in CPE is highly system-specific and must be guided by a sound understanding of metal speciation, ligand chemistry, and the underlying extraction mechanism. Careful selection and optimization of modifiers are therefore essential for achieving efficient and selective metal ion extraction, particularly in complex matrices such as those encountered in nuclear chemistry.

CPE: Role of pH

Cloud point extraction is typically performed within a moderate pH range, as highly acidic conditions can adversely affect micellar coacervation. Elevated acid concentrations tend to disrupt micellar systems due to their strong stripping and dehydrating effects, which hinder effective phase separation. Consequently, control and optimization of solution pH are essential to achieve maximum extraction efficiency (EE) [1–5]. The importance of pH control in CPE arises primarily from the pH-dependent solubility and speciation of metal ions in aqueous media. Changes in pH can alter the predominant metal species, thereby influencing their affinity toward

chelating ligands and subsequent partitioning into the surfactant-rich phase. Metal–ligand complexation is also strongly governed by pH-dependent factors, including competition from other ions, the availability of the preferred metal ion species for coordination, and the protonation–deprotonation behavior of the chelating agent, often dictated by its pK_a value [1–5]. These effects collectively determine the stability and hydrophobicity of the metal chelates formed during extraction.

To ensure reproducible and efficient extraction, the solution pH is generally adjusted and maintained using suitable buffer systems prior to the cloud point phase separation. In many cases, buffering also helps to preserve the integrity of the micellar system during heating and cooling cycles associated with CPE. Consistent control of pH before and after phase separation is therefore critical for reliable analytical performance.

Notably, recent studies have demonstrated strategies to extend the applicability of CPE to highly acidic media. Labrecque et al. [14] reported successful cloud point extraction of plutonium in 3 mol L⁻¹ nitric acid by stabilizing the micellar system through the formation of a protective bromine-rich layer around the micelles. This approach effectively shielded the micelles from acid-induced disruption, highlighting the potential for innovative micellar stabilization strategies to enable CPE under extreme chemical conditions relevant to nuclear fuel processing.

CPE: Role of electrolyte

The addition of electrolytes plays a crucial role in cloud point extraction by controlling the ionic strength of the solution, which directly influences both the cloud point temperature (CPT) of the micellar system and the overall extraction process. Various inorganic salts, particularly those containing chloride (Cl⁻), bromide (Br⁻), iodide (I⁻), nitrate (NO₃⁻), and thiocyanate (SCN⁻) ions, are known to significantly modify the micellar environment and typically lead to a decrease in CPT [1–5,15]. Electrolytes exert their effect primarily through a salting-out mechanism, wherein solvated ions compete with surfactant head groups for water molecules. This competition reduces the hydration of the surfactant, promoting earlier micellar dehydration and coacervation, thereby lowering the CPT of the micellar system. The magnitude of this effect depends on the type and concentration of the electrolyte, allowing the CPT to be systematically tuned by appropriate electrolyte selection and optimization.

In addition to modulating CPT, electrolytes can enhance extraction efficiency by promoting the incorporation of metal–ligand complexes into the micellar phase. By reducing the solubility of these complexes in the bulk aqueous phase, electrolytes effectively drive their partitioning into the surfactant-rich phase [1–5]. However, the choice of electrolyte must be made with care, as certain anions possess significant metal-complexing ability and can interfere with the desired metal–ligand interactions. Anions such as nitrate (NO_3^-), thiocyanate (SCN^-), and acetate (CH_3COO^-) are known to form complexes with various metal ions and may therefore influence extraction selectivity and efficiency [14]. Furthermore, under strongly acidic conditions ($\text{pH} < 2.5$), iodide ions can undergo oxidation to elemental iodine, which is readily solubilized in the micellar core and may adversely affect metal ion extraction efficiency [10]. Consequently, electrolyte addition is an essential but sensitive component of CPE methodology and must be implemented judiciously, with careful consideration of its concentration, chemical speciation, and potential interactions with both the metal ion and the micellar system.

CPE: Role of temperature

Accurate determination of the cloud point temperature (CPT) of a micellar system under fully optimized experimental conditions is essential for effective cloud point extraction. The CPT can be identified by simple visual observation of solution turbidity or determined more precisely using instrumental techniques such as dynamic light scattering (DLS) and viscosity measurements, which provide insight into micellar growth and phase separation behavior [1–5]. Temperature related parameters, particularly incubation time and equilibration temperature, play a critical role in achieving efficient phase separation. The incubation time must be sufficiently long to allow complete metal–ligand complexation and equilibration between the aqueous and micellar phases prior to cloud point induction [1–5]. Inadequate incubation may lead to incomplete complex formation and reduced extraction efficiency.

Surfactants exhibiting CPT values close to room temperature are generally preferred for CPE applications. This minimizes the need for external heating, which can adversely affect extraction efficiency in systems involving volatile elements such as mercury, or where metal–ligand complexation is exothermic in nature [1–5]. Elevated temperatures may also destabilize certain complexes or promote analyte loss through volatilization. Among non-ionic surfactants, Triton X-114 is the most widely employed in CPE methodologies due to its favorable

physicochemical properties, including a theoretical CPT in the range of 23–25 °C at concentrations above its CMC (Table 1). This allows efficient phase separation to be achieved under mild thermal conditions, making Triton X-114 particularly suitable for routine analytical and separation applications.

CPE: Basic equations of CPE

The performance of a newly developed cloud point extraction (CPE) methodology for a given metal ion is commonly evaluated using parameters such as percentage extraction efficiency (EE), percentage recovery, and the preconcentration factor (PF). These figures of merit provide quantitative measures of the effectiveness and analytical utility of the extraction process and are calculated using the following expressions [10]:

$$EE = \frac{C_{\text{initial}}V_{\text{initial}} - C_{\text{supernatant}}V_{\text{supernatant}}}{C_{\text{initial}}V_{\text{initial}}} \times 100$$

$$\text{Recovery} = \frac{C_{\text{SRP}}V_{\text{SRP}}}{C_{\text{initial}}V_{\text{initial}}} \times 100$$

$$PF = \frac{C_{\text{initial}}V_{\text{initial}} - C_{\text{supernatant}}V_{\text{supernatant}}}{C_{\text{supernatant}}V_{\text{supernatant}}}$$

where C_{initial} , C_{SRP} and $C_{\text{supernatant}}$ are the concentrations of respective analyte initially taken, in the SRP and in the supernatant after phase separation respectively. Similarly V_{initial} , $V_{\text{supernatant}}$ and V_{SRP} are the initial volume of the solution, volume of the supernatant phase after phase separation and volume of the redispersed SRP respectively.

The primary objective of any CPE procedure is to achieve quantitative extraction efficiency and recovery while maximizing the preconcentration factor. High EE and recovery values ensure effective transfer of the analyte into the SRP with minimal losses, whereas a high PF reflects the ability of the method to concentrate trace-level analytes into a small volume, thereby enhancing detection sensitivity and analytical performance.

Application: Environmental

The extensive use of lanthanide elements in medical, electrical, agricultural, and nuclear applications has led to their increasing release into the environment. Prolonged exposure to elevated concentrations of lanthanides can pose potential risks to human health; therefore, their

rapid and reliable determination in environmental matrices is essential for effective monitoring and mitigation [16–18]. To address this need, an offline coupling of cloud point extraction (CPE) with total reflection X-ray fluorescence (TXRF) spectrometry was developed for the simultaneous determination of lanthanides at ultra-trace levels [19]. This combined approach enabled simple, rapid, and precise determination of ten lanthanide elements whose characteristic L-emission lines exhibit a high degree of spectral overlap at low concentrations. Selective extraction of lanthanides was achieved using N,N,N',N'-tetraoctyldiglycolamide incorporated into Triton X-114 micelles, even in the presence of high concentrations of naturally abundant cations and anions. Due to the complexity of the overlapping lanthanide spectra, a multivariate partial least squares regression (PLSR) calibration strategy was employed for accurate quantification. Using this methodology, ten lanthanides, namely La, Nd, Sm, Eu, Gd, Tb, Dy, supramolecular complex with Th(IV) via a cation-exchange mechanism. Under optimized conditions, the method exhibited an extraction efficiency of $(98.9 \pm 0.2)\%$ and a recovery of 99%, with a preconcentration factor of 91. These favorable figures of merit resulted in effective quantitative enrichment of Th(IV) into the surfactant-rich phase (SRP), which was subsequently analyzed directly using TXRF spectrometry. The microlitre-scale sample Ho, Tm, and Lu, were simultaneously determined over a concentration range of 10 to $(5 \times 10^3) \mu\text{g L}^{-1}$. Method validation was carried out by analyzing three certified reference materials (CRMs), NASS-7 seawater, SRLS-6 river water, and NIST 1640a natural water, using the standard addition approach, yielding relative standard deviations of $\leq 10\%$. A schematic representation of the overall analytical workflow is shown in Figure 2.

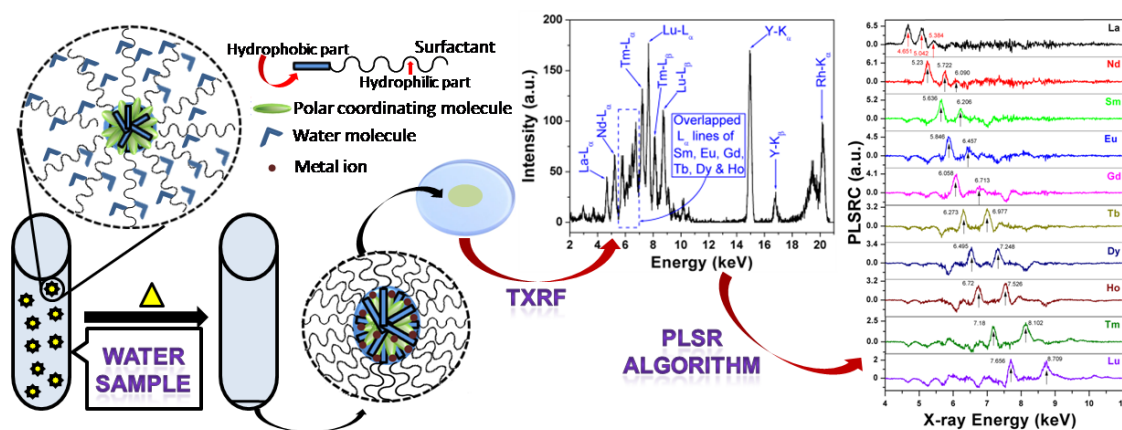


Fig. 2. Complete illustration of the CPE of 10 Ln's followed by their determination by PLSR calibration assisted TXRF spectroscopy.

Thorium presents a distinct environmental concern due to its combined chemical and radiological toxicity [20,21]. Anthropogenic activities such as mining and processing of thorium-bearing minerals, as well as its industrial applications, contribute to increased human exposure. Thorium compounds are recognized as potential occupational carcinogens, and their low solubility in natural waters necessitates ultra-trace level determination of Th(IV). This requirement is further complicated by the presence of chemically similar and ubiquitously occurring elements such as uranium and lanthanides, along with abundant alkali, alkaline earth, and transition metal ions. To address these challenges, a selective CPE method for Th(IV) was developed using a bis(phosphoramidate) ligand. The extraction parameters were systematically optimized to achieve quantitative recovery of thorium. At pH 4, the ligand, in conjunction with sodium dodecyl sulfate in the micellar medium, was found to form a requirement of TXRF makes it particularly well suited for direct analysis of the SRP without compromising the pre-concentration factor. Furthermore, oxidative pyrolysis of the SRP prior to TXRF analysis significantly improved the thorium detection limit from 0.9 to 0.1 $\mu\text{g L}^{-1}$ [22]. The analytical methodology was validated through the analysis of certified reference materials and real environmental samples using spike addition, yielding a maximum relative precision of 2%. A schematic illustration of the proposed CPE mechanism for Th(IV) preconcentration is presented in Figure 3.

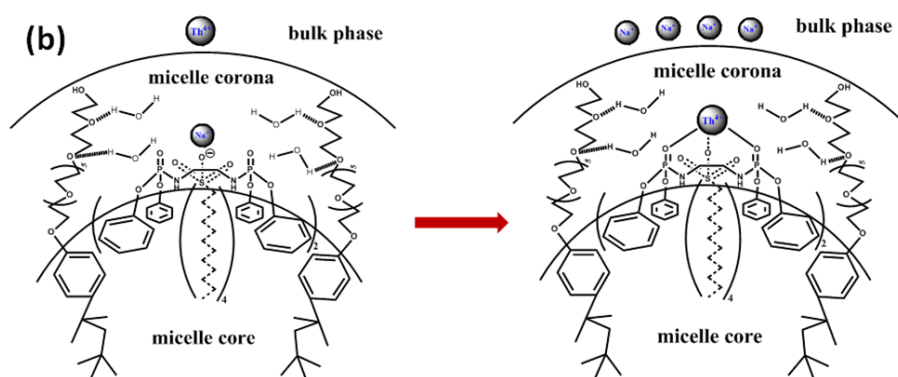


Fig. 3. Proposed CPE mechanisms of Th(IV) in absence of SDS (a) and in presence of SDS (b).

Application: Nuclear industry

The determination of trace-level impurities in nuclear fuel materials is a critical component of the chemical quality control (CQC) program, as such impurities can significantly affect fuel performance, reactor safety, and neutron economy [23–25]. Among these, the separation and

determination of boron in uranium-based nuclear fuels pose a particular analytical challenge. This difficulty arises primarily from the highly acidic nature of fuel dissolution media, which adversely affects micellar coacervation during cloud point extraction. Elevated acidity increases the cloud point temperature (CPT) of the micellar system, thereby necessitating heating, which can lead to unavoidable losses of boron due to the formation of volatile species. To overcome these limitations, a novel CPE-assisted colorimetric method was developed for the determination of trace amounts of boron in uranium-based fuels. In the proposed approach, a 1:1 mixture of 2-ethylhexane-1,3-diol (EHD) and curcumin was dispersed in Triton X-114 micelles to selectively extract boron from acidic uranium matrices [26]. Several compounds were investigated as potential micelle surface modifiers to stabilize the micellar system under high-acidity conditions. Among them, bromine water (Br_2) was found to be uniquely effective, not only significantly reducing the CPT from approximately 80 °C to 42 ± 2 °C, but also enabling quantitative recovery of boron ($\geq 95\%$). The optimized CPE procedure allowed efficient extraction of boron from uranium matrices in 2.0 mol L⁻¹ HCl, rendering it suitable for routine chemical quality assurance of uranium-based nuclear fuels. The boron–EHD–curcumin complex exhibited a high molar extinction coefficient of 4.75×10^5 L.mol⁻¹.cm⁻¹ at a wavelength maximum of 458 nm in N,N-dimethylformamide medium. The method demonstrated a linear dynamic range of 10–150 ng mL⁻¹ with a detection limit of 0.8 ng mL⁻¹. Validation was carried out using three in-house uranium working reference materials, and successful determination of trace boron was demonstrated in two uranium dioxide and two metallic uranium samples. The relative standard deviation of the method was found to be in the range of 3–5%, confirming its precision and robustness. A schematic representation of the boron CPE process is shown in Figure 1, while the effect of increasing boron concentration on the extraction behavior is illustrated in Figure 4.

In another application relevant to nuclear fuel reprocessing, a novel cloud point back-extraction (CPEback) method was developed for the quantitative stripping of uranium(VI) from organic radioactive solutions into an aqueous phase [27]. This unprecedented approach exploited the difference in nitrate ion concentration between the micellar phase and the bulk aqueous phase to shift the uranium distribution coefficient in favor of the aqueous medium. The inherently monophasic nature of cloud point extraction, combined with the strong complexing ability of oxalic acid toward the uranyl ion in the aqueous phase, enabled quantitative uranium recovery in

a single step (Figure 5). The parameters governing the CPEback process were optimized through a systematic cross-optimization strategy, and the recovered uranium was quantified using biamperometric titration. The method achieved a back-extraction efficiency of $(99.6 \pm 0.2)\%$ and a recovery of 99.2%. Importantly, this process converted large volumes of organic radioactive waste into small volumes of a less hazardous organic phase characterized by high viscosity and negligible vapor pressure. Application of the method to two real organic radioactive solutions with volumes of approximately 17 and 165 mL resulted in uranium recoveries of 99.0% and 98.2%, respectively. The residual organic phase, containing the extractant and non-ionic surfactant, could be readily stored or reused, highlighting the method's potential for sustainable radioactive waste management.

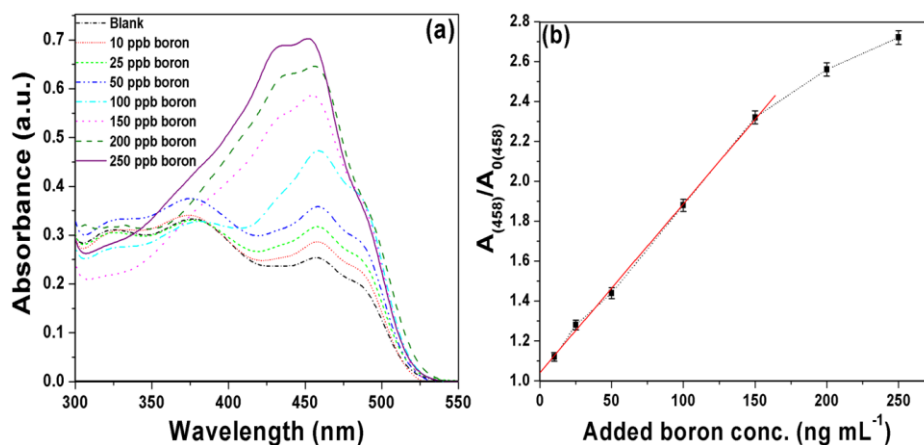


Fig. 4 (a) Effect of adding increasing B concentrations on the absorption intensity of SRP in presence of Br₂ and (b) plot of $A_{(458)}/A_{0(458)}$ vs concentration of boron and calibration spectrum in the range of 10-150 ng mL⁻¹. [HCl] = 2.0 mol L⁻¹, [TTX-114] = 25 mmol L⁻¹, [EHD + Curcumin] = (0.25 + 0.25) mmol L⁻¹, [Br₂] = 0.1 mol L⁻¹, Text. = 4-5 oC, and Tphs. sep. = 40 oC. (Black dotted line = experimental fit and red solid line = linear regression fit)

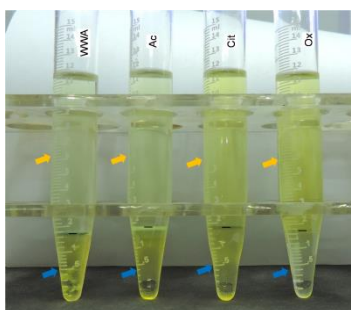


Fig. 5. Visual representation of the bulk aqueous phase (orange arrow) and SRP (blue arrow) after completion of CPE without weak acid (WWA), with acetic acid (Ac), citric acid (Cit) and oxalic acid (Ox). The black horizontal line represents the phase separation.

Conclusions

Cloud point extraction has emerged as a highly efficient, versatile, and environmentally benign separation and preconcentration technique with broad applicability in environmental analysis and the nuclear industry. Its inherently monophasic extraction mechanism imparts higher efficiency compared to conventional liquid–liquid extraction (LLE), while significantly reducing the use of toxic organic solvents. These advantages position CPE as a greener and more sustainable alternative to LLE in several critical areas of nuclear and environmental science.

A comprehensive understanding of the roles of surfactants, metal-coordinating ligands, phase modifiers, electrolytes, pH, and temperature is essential for the rational design of robust CPE methodologies. Careful optimization of these parameters enables selective and quantitative extraction of target metal ions even from chemically complex and highly acidic matrices. The ability of CPE to achieve high extraction efficiencies, recoveries, and preconcentration factors makes it particularly suitable for the determination of trace- and ultra-trace-level metal ions.

The application of CPE to environmental analysis demonstrates its effectiveness in the preconcentration and quantification of lanthanides and actinides, thereby facilitating their reliable detection at low concentration levels. Such capability is important not only for mitigating the potential chemical and radiological hazards associated with these elements, but also for their identification and assessment during deep geological surveys, where accurate trace-level determination is essential for resource evaluation.

In the nuclear industry, CPE-based methodologies address key challenges in chemical quality control and radioactive waste management. The successful application of CPE for trace boron determination in uranium-based nuclear fuels represents the first work of its kind and highlights the method's potential to replace existing LLE-based procedures in routine CQC of nuclear materials. Furthermore, the unprecedented application of cloud point back-extraction for the quantitative recovery of U(VI) from waste organic streams underscores the remarkable extraction capability of CPE and its utility in converting hazardous organic radioactive solutions into safer and more manageable forms.

Overall, the studies discussed in this work clearly demonstrate that cloud point extraction is not merely an alternative but a powerful and forward-looking separation strategy. Continued advances in micellar chemistry, ligand design, and system modification are expected to further

expand the scope of CPE, reinforcing its role as a key analytical and process tool in nuclear chemistry, environmental monitoring, and sustainable separation science.

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About the Authors



Dr. Abhijit Saha joined Bhabha Atomic Research Centre, Mumbai, India, as a scientific officer in 2012 after completing a one-year orientation course from 6th batch of IGCAR Training School (equivalent to 55th batch of BARC Training School). His current research area includes a synthesis of surface functionalized nanoparticles, synthesis of organic ligands, cloud point extraction (CPE), development of different separation methodologies for nuclear application, direct solid analysis by ETV-ICP-OES. To date, he has published about 25 research papers in peer-reviewed international journals. He was honored with the Prof. H. J. Arnikaar Best award in the year 2019.



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Hybrid Polymer-Supported Graphene Oxide Composites: Next-Generation Adsorbents for Selective U and Zr Separation

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Summary

Separation chemists have identified graphene oxide (GO) as a promising solid-phase platform for metal ion recovery, particularly for ions of strategic relevance to the nuclear industry, owing to its capacity to host a diverse array of functional groups. Monolayer GO features sp^2 -hybridised carbon domains analogous to graphene sheets, interspersed with a disrupted sp^3 -hybridized framework arising from extensive oxidation and the concomitant introduction of oxygen-containing functionalities. The coexistence of these functional groups with an extended delocalized π -network permits post-synthetic functionalization with a variety of ligating moieties and exerts a decisive influence on the chemical reactivity, surface polarity, and overall physicochemical behavior of GO. To circumvent limitations associated with its finite lateral dimensions, colloidal nature, and handling, GO has been incorporated into composite adsorbent architectures, either embedded in polymeric matrices or immobilized on inorganic supports such as iron oxides. GO-polystyrene composite beads, for instance, couple the high specific surface area and rich surface chemistry of GO with the enhanced particle size, mechanical robustness, and processability imparted by the polystyrene framework. Likewise, magnetic GO-based composites enable magnetically assisted separation and recovery of the sorbent phase, thereby addressing constraints related to solid-liquid separation and recyclability. Such GO-based composite adsorbents have been successfully deployed for the selective uptake and separation of U and Zr from aqueous streams pertinent to nuclear fuel cycle and waste management.

Introduction

The recovery of strategic and precious metal ions such as U and Pd from aqueous solutions is of significant importance in nuclear fuel cycle operations, particularly with respect to the re-use of

valuable resources and precious metal recycling [1]. The challenging separation methods involved in recovering these metal ions to the desired purity levels constitute a crucial aspect of metal ion separation. Most importantly, exploring options to separate U from alternative sources other than conventional routes, through solid-state separation approaches exploiting materials that are highly selective for its separation, is essential. Whilst Zr is another element of importance in the nuclear industry, projected to be used along with U and Pu for proposed future U-Pu-Zr fuels in advanced fast breeder nuclear reactors, its separation from nuclear waste also assumes significance [2,3]. The above challenges can be addressed through the selective removal of these elements in the presence of complex aqueous feed solutions and chemically similar metal ions that may co-separate with them. The conventional separation techniques may not be suitable for selective separation purposes, considering the desired purity levels and owing to other inherent complexities such as poor selectivity, secondary waste generation, and poor performance at trace concentrations. In this context, advanced separation systems utilizing solid-phase adsorbents with large surface properties, such as graphene oxide have emerged as promising next-generation adsorbent materials. In addition to their unique surface characteristics, their tunable properties, which facilitate the preparation of metal-ion-specific adsorbents suitable for target-specific removal, make these materials an attractive choice for separation applications [4–6].

Graphene, a monolayer allotrope of carbon, and its oxidized sibling graphene oxide (GO) are the materials with versatile applications that drawn special focus in metal ion separation because of their fascinating properties, notably high surface area, flexibility to incorporate different functionalities and excellent chemical stability [5]. Compared to graphenes, GO allows facile surface modification and incorporation with diverse ligands makes them attractive choice for metal ion separation. Usually, a GO-based matrix is prepared out of the graphite phase, and therefore, a typical GO matrix consists of either a monolayer or has few of layers of functionalized graphene. Hummers' method or its modification is a well-recognized chemical procedure for preparing GOs in large quantities amenable for industrial level scaling up. It has also been known that oxidation of graphite matrix resulted in mono-layered graphene or graphitic oxide having fewer layers. The oxidation process also results in the formation of several functional groups on the matrix, such as carboxyl, sulfonyl, hydroxyl and epoxy groups.

This work leverages GO's adaptability to develop practical adsorbents for nuclear remediation. Amine-functionalized GO-polymer composites and pristine magnetic GO exploiting carboxylate chelation through rapid magnetic recovery both effectively target U extraction from seawater-like solutions with minimal interference from competing ions. Complementing this, amide-functionalized GO-polystyrene composites demonstrate exceptional selectivity for Zr extraction from PUREX-origin acidic waste, overcoming challenges posed by high acidity and chemically similar metal ions through synergistic amide-polymer interactions.

Preparation of GO

The Hummers' method of GO preparation involves oxidation of graphite [4–6]. A typical preparative procedure involves, oxidation reaction of approximately 5 g of graphite powder using potassium permanganate (20 g), along with sulphuric acid (450 mL) and orthophosphoric acid (50 mL) (acid mixture was taken in 9:1 ratio). The reaction was left to continue for 6 hours with stirring at a constant temperature maintained at 40°C. Ice cold water was then added slowly to the above mixture. Finally, 10% hydrogen peroxide was added until golden brown GO was

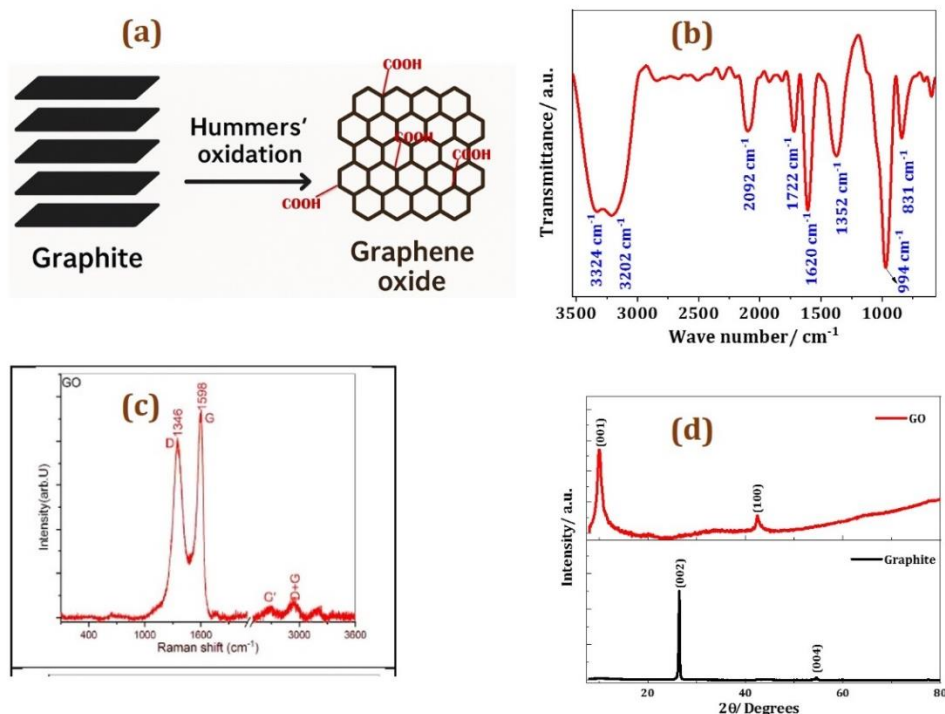


Fig. 1. Hummers' oxidation based preparation of graphene oxide from graphite powder (a) FTIR spectrum (b) Raman spectrum (c) and XRD (d) of GO

observed, indicating the elimination of manganate ions by reduction. The GO produced was then filtered using vacuum flask washed with dilute hydrochloric acid and dried at 60°C under vacuum. Preparative scheme of GO is shown in Fig. 1 (a).

The standard methods for characterizing GO include infrared and Raman spectroscopy, which reveal its characteristic functional groups and defect structures. While XRD confirms the formation of the GO framework, morphological details are obtained via SEM and TEM. The FT-IR and Raman spectra, as well as the XRD pattern of the GO prepared above, are shown in Fig. 1 (b–d). The multilayered GO exhibits signature FT-IR peaks at 1722 and 1620 cm^{-1} , corresponding to the carbonyl (C=O) stretching associated with carboxylic acid groups. The peak at 994 cm^{-1} indicates the possible presence of epoxy groups or the S=O stretching frequency of sulfonic acid groups; the latter may be introduced during synthesis since sulfuric acid is used as a precursor. The O-H stretching vibrations associated with hydroxyl and sulfonic acid groups were observed as a broad band spanning 3500-3000 cm^{-1} . Additionally, the feature at 831 cm^{-1} corresponds to the deformation vibrations of epoxides (C-O-C). The Raman spectrum of GO displays peaks at 1346 cm^{-1} and 1598 cm^{-1} , designated as the D (defect) and G (graphitic) bands, respectively. The XRD pattern shown in Fig. 1(d) reveals a signature peak at approximately 12°, corresponding to the (001) plane, which confirms the formation of GO. It also indicates a residual graphite peak at 25°, corresponding to the (002) plane. In essence, the combined FT-IR, Raman, and XRD data confirm the successful synthesis of few-layered GO from graphite. The presence of oxygen-rich functional groups including carbonyl, hydroxyl, and epoxy groups, as well as potential sulfonic acid residues is evidenced by distinct vibrational peaks. Structurally, the Raman and XRD analyses highlight the transition from a bulk graphitic state to a defective, layered framework, characterized by the signature (001) crystalline plane and a high defect density [4,6].

Composite adsorbents based on GO

The practical utility of GO for metal ion adsorption is challenged by its fine size. The fine powders of GO can choke columns. Therefore, adopting GO in a separation system requires an engineered adsorbent with a larger particle size to prevent column choking and to minimize back pressure. One convenient method to achieve this is the development of a polymeric-GO hybrid material. For the separation of U, we developed an amine-functionalized GO-polystyrene

composite material. Likewise, for Zr removal from acidic solutions, an amide-functionalized GO–polystyrene composite was developed. These hybrid materials are prepared by unique *in situ* polymerization in the presence of functionalized GO. Additionally, the GO architecture was adapted to a magnetic solid-phase adsorption system: another class of hybrid adsorbents in which metal ion separation can be achieved without the need for column-based separation systems. Compared to conventional column-based systems, these magnetic adsorption systems can be implemented in batch mode for metal ion recovery, and the presence of a paramagnetic framework facilitates efficient disengagement of the adsorbent from the aqueous phase.

GO polystyrene composite adsorbent

As indicated, GO's practical utility was explored by making a hybrid adsorbent along with polystyrene matrix. For this purpose, the functionalized GO selective for the particular metal ion separation was prepared initially and was incorporated to the polymer matrix by in-situ Pickering emulsion polymerization. The major highlight of these types of composite material is that they can be utilized for larger-scale column type operations wherein GO's finite size is a limitation.

The amine-functionalized GO was prepared by dispersing 6.5 g of GO in ethanol via sonication for 15 minutes, followed by the addition of ~40 mL of diethylenetriamine (DETA). The mixture was then heated at 80°C for 2 hours. The hybrid properties of these two materials are expected to exhibit superior U uptake compared to previously reported adsorbents of a similar nature. Furthermore, the micrometer-sized polystyrene beads observed in the SEM images (Fig. 2) suggest the potential for employing these materials in large-scale column studies.

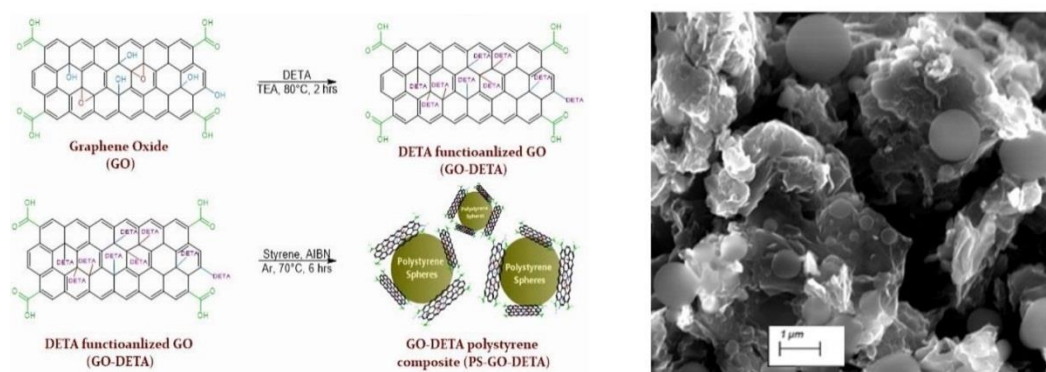


Fig. 2. Synthesis scheme adopted for the preparation of PS-GO-DETA composite (left) SEM image of GO-amine functionalized polystyrene beads (right).

The adsorbent, amine functionalized GO-polystyrene composite was prepared by in-situ polymerization of styrene in the presence of the amine functionalized GO. Pickering emulsion polymerization is a simple but efficient way of preparing polymers-GO template. A typical synthetic procedure consist of dispersing 0.5 g of GO-DETA in 100 mL distilled water (5 mg mL⁻¹), with 25 mL of styrene in presence of 2,2'-azobis(isobutyronitrile), AIBN (10 mg) to initiate polymerization. The reaction was left to continue for 6 hours at 70°C. The obtained product was filtered and washed with acetone multiple times to remove styrene monomers. The as prepared PS-GO-DETA is employed for further studies. The reaction scheme followed for preparation of the adsorbent is summarized in Fig. 2. The resulting amine-functionalized GO-polystyrene composite was subsequently evaluated for its U removal efficiency from aqueous solutions.

The U adsorptive features of the above adsorbent in the presence of metal ions that are present in sea water condition have been tested, and the data is shown in Fig. 3. It was observed that V, Mg, Si and Sr were extracted to a lesser extent (less than 5 %), whereas Ni, Cu, Co, Na and Ca were adsorbed to less than 30 %. U adsorption to the resin was more than 90 %. Therefore, it can be expected that PS-GO-DETA can remove U from sea water like solutions with minimal interference from other ions.

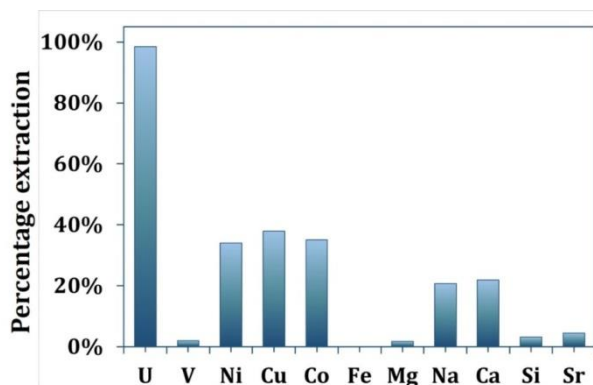


Fig. 3. Extraction of U and other ions present in sea water. Sea water has been spiked with U for the separation.

Another polymeric-GO composite adsorbent having amide functional group on GO matrix was prepared for the separation of Zr from PUREX originated acidic waste that could originate during the reprocessing of U-Pu-Zr based fuel through aqueous reprocessing route.

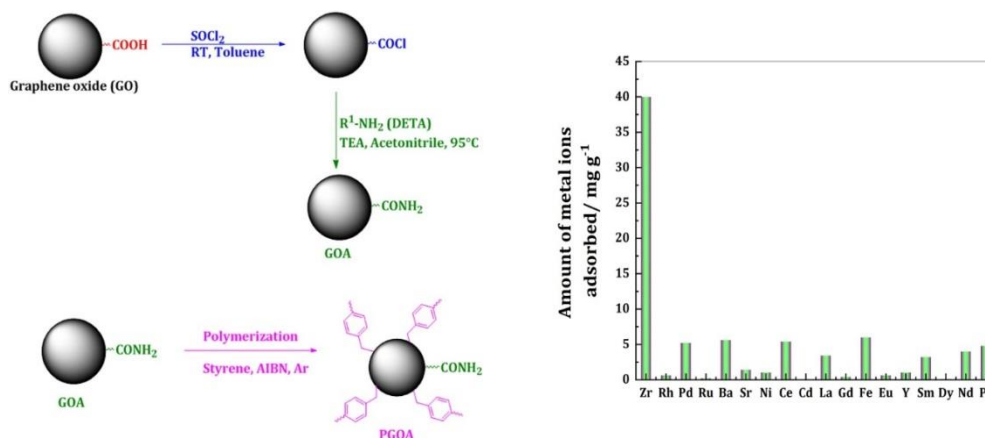


Fig. 4. Synthetic scheme for the preparation of PGOA (left) Loading behavior of various elements present in the fast reactor simulated high level waste, corresponding to 155 GWd/Te burn up (right).

The synthesis of GOA involves chemical reaction of GO (3 g) with thionylchloride (3 mL) in the presence of chloroform (50 mL) to generate GO-COCl, and then reacting the resultant GO-COCl (1.5 g) with DETA (4.5 mL) in the presence of triethylamine (TEA). The produced amide was purified by washing it with ethanol, and the GOA particles were dried under vacuum (at 50 °C) for solvent evaporation (30 min) before use. The adsorbent, amide functionalized GO-polystyrene composite (PGOA) was also prepared by the Pickering emulsion polymerization as discussed above. The chemical functionalization of amide moiety to GO surface (GOA, as shown in Fig. 4) was performed by conversion of the acidic functional groups of GO to oxy chlorides and subsequent introduction of amine to make them amide derivative (See Fig. 4).

For the Zr separation studies, the feed solution considered was acidic that could be generated from the reprocessing of U-Pu-Zr or U-Zr generated PUREX raffinate, where the presence of Zr may limit the efficacy of trivalent removal by TRUEX. In this connection, the ability of PGOA to adsorb Zr from aqueous phase was examined in a variety of acidic solutions ranging from 1 to 7 M nitric acid. In all nitric acid concentrations ranging from 1 to 6 M, Zr adsorption was high and it partially decreases above 6 M. Zr has an increased tendency to hydrolyze and polymerize depending on the acidity, and the resultant Zr aqueous chemistry is complicated. The Zr-uptake capability of PGOA in the presence of other elements present in the proposed feed (High-Level Liquid Waste; HLLW generated from U-Pu-Zr fuel) has been studied using a simulated waste composition corresponding to 80 GWd/Te [5]. Fig. 4 also shows adsorption of different elements to the adsorbent. The amount of metal ions adsorbed was

calculated from the difference in the concentrations of the metal ions before and after contacting with PGOA. It can be concluded that even in the presence of other elements a significant portion of Zr ($\sim 40 \text{ mg g}^{-1}$) was loaded to PGOA. Approximately 5 mg g^{-1} each of Fe, Ba, Nd and Ce were loaded to PGOA, whereas loading of other elements were quite insignificant. Results of the study pointed towards the feasibility of employing PGOA for the separation of Zr from the PUREX raffinate (HLLW) of U-Pu-Zr derived fuel waste, so that the possible complications in TRUEX anticipated due to the presence of Zr can be averted. Overall, PGOA can be endorsed as an efficient solid-state adsorbent for the bulk removal of Zr from the PUREX raffinate generated from U-Pu-Zr metallic fuel.

Magnetic graphene oxides

Magnetic solid phase separation is a novel solid-phase technique wherein separation can be achieved in batch mode of operation, and the adsorbent can be separated from the liquid phase by the application of an external magnet. A typical example of magnetic solid phase separation is depicted in Fig. 5. In a magnetic solid phase separation system, the role of the magnetic moiety (Fe_3O_4) is only to dictate the consolidation of the adsorbent after the chelating interaction of the ligand with the metal ion. Therefore, metal ion separation can be optimized by optimizing the ligand component. Separation of U from seawater-like solutions was attempted using a hybrid adsorbent based on GO and Fe_3O_4 . Unlike previous attempts, pristine GO was investigated as such for ion-exchange-based separation of U [7].



Fig. 5. Images of prepared MGO, dispersed MGO, and magnetic separated of MGO in the presence of external magnet.

As discussed earlier, the Hummers' chemical exfoliation reaction produces several functional groups (viz. COOH , SO_3H , OH , etc.) on GO, which can be exploited for metal ion

separation. Optimizing the surface functional groups on GO is a key aspect when employing it for adsorption applications. By suitably optimizing synthetic methods, GO predominantly consisting of carboxylic acid groups can be prepared. MGO was prepared by co-precipitation of, involving alkaline hydrolysis (using ammonia, 800 mL) of a mixture of FeCl_3 (0.8 M, 10 g) and FeSO_4 (0.4 M, 7.3 g) in millipore water (250 mL), in the presence of GO. For this purpose, GO powder was dispersed in the alkaline solution during the co-precipitation process, by refluxing at 85°C for two hours. The MGO particles formed were separated from the mixture and washed several times with de-ionized water, isopropanol and acetone. The MGO particles were dried in air overnight before use. A permanent magnet (1 tesla, cylindrically shaped, of 100 mm length and 15 mm diameter) was used for the physical separation of magnetic particles from liquid phase. Fig. 5 shows the images of the magnetic-GO hybrid adsorbent, its dispersed form in aqueous phase, and the consolidation of the adsorbent when an external magnet is kept close by.

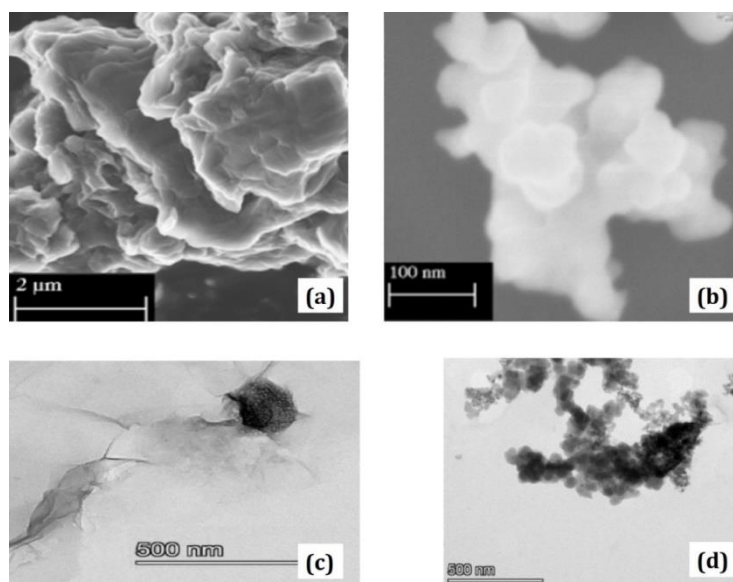


Fig. 6. SEM images of GO (a) and MGO (b). TEM images of GO (c) and MGO (d)

When GO and Fe_3O_4 are both present in a single material, wherein they individually also exhibit a layered framework, the combined hybrid material should exist as interlayers of GO and Fe_3O_4 [6]. The SEM and TEM images confirm this by showing the interwoven layers of GO and Fe_3O_4 . The SEM images indicate that the samples are powder agglomerates and the high resolution image of MGO shows the particles as flat agglomerates, as compared to GOs (Fig. 6 (a) and 6(b)). TEM images of MGO (Fig 6 (c&d)) show Fe_3O_4 nanoparticles dispersed in the

GO layer, and therefore it assists in faster disengagement of the MGO from the aqueous solutions. In the MGO, the spherical Fe_3O_4 particles are embedded in the interlayers of GO. The graphene wrapping around Fe_3O_4 has enhanced stability in the MGO-based adsorbents. Besides assisting the physical separation of the adsorbent from the aqueous phase (fast separation through magnetic field), Fe_3O_4 has no role in the adsorption process.

Adsorption of U to MGO shown in Fig. 7. The adsorbent exhibited higher adsorption of U above pH 5, with adsorption capacity above 120 mg g^{-1} . The adsorbent was also tested for its suitability of U removal from sea water like conditions. The amount of U adsorbed onto MGO was determined in the presence of some of the interfering cations, such as Fe, V, Zn, Cu, Ni, Co, Cs and Mg and the results of which are shown in Fig. 7. It can be that except in the presence of V, the reduced U adsorption in the presence of other interfering ions was only 25 % in case of Fe. Reduction in adsorption of U in presence of other elements such as Zn, Cu, Ni, Co, Cs, and Mg was within 5 %. U adsorption was reduced from 120 mg g^{-1} to 65 mg g^{-1} when $200 \text{ }\mu\text{g/mL}$ of V was present alongside $100 \text{ }\mu\text{g/mL}$ of U. The reduction in the adsorption of U in presence of V could be due to the presence pentavalent oxidation state of V exhibiting significant competition to U^{6+} . Moreover, the actual concentration of V in seawater is 5 to 6 times lower than that of U, while in the present study concentration of V was twice that of U. Overall, efficiency of MGO in selectively separating U even in the presence of interfering cations is highly superior.

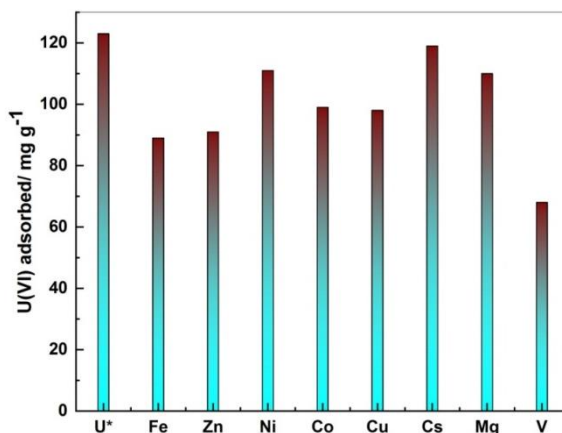


Fig. 7. Adsorption of U to MGO in the presence of interfering (Fe, Zn, Ni, Co, Cu, Cs, Mg, V) ions. Adsorbent: 100 mg of MGO. Aqueous phase: 100 mg L^{-1} U present in sodium acetate medium at pH 6. 200 mg L^{-1} of the interfering ions is present as the chloride or nitrate salt. $T=298 \text{ K}$. Duration = 6 hours. The asterisk on U indicates that the U was adsorbed in the absence of other ions.

Conclusions

The monolayered structure of GO provides exceptional adaptability for incorporating diverse ligands, positioning it as a promising platform for solid-phase adsorption in nuclear waste remediation. However, its nanoscale dimensions limit large-scale applications; this challenge is effectively addressed by integrating GO into micrometer-sized polystyrene beads or magnetic moieties, which prevent column clogging while enhancing mechanical stability and handling.

An amine-functionalized GO and the pristine magnetic GO excels in U removal from seawater-like solutions, where optimized carboxylate/amine groups drive strong chelation, and magnetic components enable rapid phase separation. Similarly amide-functionalized GO-polystyrene composites demonstrate superior selectivity for Zr extraction from PUREX-origin acidic waste, achieving high efficiency with minimal interference from co-existing metal ions, thanks to the synergistic interplay of amide ligands and the polymer matrix. Similarly,

Overall, GO's surface properties can be precisely tuned through modified Hummers' oxidation and targeted ligand grafting (amines for U and amides for Zr), unlocking metal-ion-selective adsorbents that bridge laboratory efficacy with practical deployment in complex aqueous matrices.

Acknowledgements

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Dr. N. Ramanathan serves as Head, Actinide Chemistry and Separation Science Section, MC&MFCG, and Associate Professor HBNI. He joined IGCAR, Kalpakkam, in 2002 post-45th batch of BARC training school. His research spans development of materials for metal ion separation for back end of nuclear fuel cycle, novel hybrids like graphene oxides and magnetic materials for advanced separation, and analytical methods related to nuclear reprocessing. He also probes molecular conformations, transient species (free radicals), novel gas phase reactions and unconventional interactions (hydrogen, halogen, tetrel, pnictogen bonding) using matrix isolation infrared spectroscopy. He has 125 peer-reviewed publications and 100 conference publications to his credit.

Development of Purification Technologies for Clean & Safe drinking Water

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Introduction

Worldwide availability of fresh water for industrial needs and human consumption are considered as major obstacles to achieve sustainable development [1]. The water demand in the country is increasing fast due to rapid population growth, accelerated urbanization, progressive increase in the demand of water for irrigation, expanding industrialization, and improving life standards. Rapid industrial expansion- an essential driver of economic transformation under initiatives like ‘Make in India’ and ‘Atmanirbhar Bharat’—will push water demand even higher in the coming decades. But the freshwater resources are under severe stress, as they are limited in quantity and increasingly degraded by domestic, agricultural, and industrial pollution. Additionally, groundwater contamination with salinity, fluoride, arsenic, iron, nitrate and microbial pollutants is widespread in many rural and peri-urban areas, rendering water unsafe for drinking and domestic use. Heavy metals contamination viz. uranium, barium, manganese, zinc etc are also found in ground water in certain regions of India.

Against this backdrop, desalination, water purification, and wastewater reuse technologies have emerged as critical tools to augment potable and process water supplies [2-3]. India has vast coastlines suitable for sea water desalination, extensive inland regions with brackish aquifers, and significant volumes of wastewater that can be reclaimed and reused. The purified water from waste effluents can be treated and reused for irrigation, cooling water and other non-potable industrial applications, thereby reserving high-quality water for domestic use for sustainable water management. Government programs such as Jal Jeevan Mission (JJM), Atal Mission for Rejuvenation and Urban Transformation (AMRUT 2.0), and initiatives under National Water Mission are already encouraging innovations in this direction.

In accordance with DAE’s mandate of ‘Atoms in the Service of the Nation’ for societal application, BARC has developed desalination and water purification technologies for a wide

range of applications which are commercially viable, energy-efficient, and environmentally compatible technologies tailored to local hydro-geological and socio-economic conditions. The developed technologies include (a) sea water desalination using thermal processes and RO membranes for coastal areas, (b) brackish water RO in villages for producing safe drinking water and (c) nanofiltration (NF) and ultrafiltration (UF) membrane-based water purification for the removal of hardness of water, microorganisms, turbidity & toxic contaminants for producing safe drinking water for domestic/community use, (d) ZLD desalination system for management of brine in a sustainable and environment friendly way.

Desalination Technologies

Desalination refers to the process by which pure water is recovered from saline water using different forms of energy. Saline water is classified as either brackish water or seawater depending on the salinity and water source. Desalination produces two streams - freshwater and a more concentrated stream (brine). The two main commercial desalination technologies are those based on thermal and membrane processes.

Thermal Desalination

Multi Stage Flash (MSF)

BARC has developed MSF technology based on long tube design requiring less energy consumption as compared to conventional cross tube MSF. Based on the experience gained from the 15 KLD desalination experimental facility and 425 KLD MSF pilot plant at Trombay, a 4.5 MLD MSF unit was designed and built as a part of the 6.3 MLD Hybrid MSF-RO plant.



4.5 MLD MSF Plant at NDDP, Kalpakkam coupled to MAPS

Multi-Effect Distillation -Thermo Vapour Compression

BARC has successfully installed & commissioned 240 m³/d capacity MED-TVC, based on horizontal falling film evaporation along with thermal vapour compression. MED-TVC has successfully demonstrated production of low conductivity ($\sim 2 \mu\text{S/cm}$) distilled water directly from seawater. BARC has also done the scaleup design for 1000 m³/d capacity MED-TVC plant. The know-how of the 1000 m³/d capacity MED-TVC plant has been transferred to private entrepreneurs for commercialization and deployment [4]. BARC has set-up a 2x250 KLD MED-TVC plant at OSCOM, which is presently under regular operation, and meeting OSCOM process water requirements.



240 m³/day capacity MED-TVC plant at BARC



Pilot scale 1 ton/day Prototype FD unit

Low temperature Freeze Desalination

Freeze Desalination (FD) is a low temperature thermal desalination technique based on freezing of saline water to form ice crystals leaving behind the concentrated brine. FD offers a promising

alternative to traditional desalination methods due to its lower energy consumption as freezing water requires roughly 7 times less energy than the evaporation. Additionally, cold temperature used in the FD process minimize the corrosion and scaling problem, allowing the use of low-cost materials. However, availability of low temperature heat sink and inherent limitations such as incomplete removal of salt from the ice have hampered widespread adoption of FD. BARC has designed and commissioned a pilot scale 1 ton/day Prototype Freeze Desalination unit using refrigeration circuit and produces ice on continuous basis. Unit has demonstrated production of water from saline water on continuous basis by melting the ice produced.

Sea water Reverse Osmosis (SWRO)

A typical RO desalting plant consists of three sections, namely pretreatment section, membrane section and post treatment section. Conventional pretreatment section typically consists of particulate filtration, micron filtration and chemicals additions. Nowadays, ultrafiltration membranes are replaced conventional pretreatment in most of the desalination plants. Membrane section consists of membrane elements housed in pressure vessels through which pretreated saline water is passed under pressure in excess of its osmotic pressure with the help of a high-pressure pump coupled with energy recovery device. The post treatment section consists of lime treatment for pH correction and chlorination for disinfection as required to meet public health standards and to make the water non-corrosive to the water distribution systems. BARC has set-up a 4.5 MLD SWRO plant at OSCOM, Odisha which is under regular operation. The 4.5 MLD SWRO plant is meeting OSCOM potable water requirements and also a part of its delivered at nearby Matikhalo village.



*4.5MLD SWRO Desalination Plant at
OSCOM, Orissa*



*1.8MLD SWRO Desalination Plant at NDDP,
Kalpakkam*

Technologies and Products Developed for Water Purifications

On-line Domestic Water Purifier Based on Ultrafiltration Polysulfone Membrane

BARC has developed the “point of use” online domestic ultrafiltration (UF) membrane-based devices with typical features such as affordability, minimal maintenance, non-requirement of the power source, minimal wastage of water (almost 100% recovery) etc. This device is based on polysulfone UF membrane in cylindrical, candle like configuration. It is an on-line device for purification of domestic drinking water which is capable of removing microorganisms, suspended solids, colloids, colour, odour and organics. This technology has been transferred to several private entrepreneurs for commercialization and deployment [5].

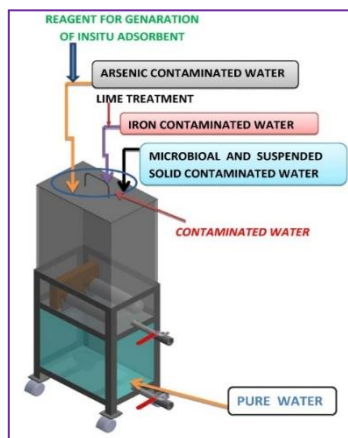


Products manufactured & marketed by BARC licensees

Nanocomposite Ultrafiltration Membrane Device for Domestic Drinking Water:

Purification with respect to Arsenic, Iron and Microbial Contaminations

The combo domestic water purifier device is made of polysulfone based nanocomposite ultrafiltration membrane in cylindrical configuration. The combo domestic water purifier device is made of polysulfone based nanocomposite ultrafiltration membrane in cylindrical configuration. The device stand alone can remove suspended materials and microorganism with bacteria removal efficiency: 99.99 % and also it can remove arsenic contamination by an in situ generated sorbent with simple addition of two pre-formed reagents and subsequent membrane filtration to provide product water with arsenic contamination < 10 ppb as per WHO standard. The device can remove iron contamination by addition of slaked lime and subsequent membrane filtration to provide product water with iron contamination < 0.3 ppm as per WHO standard. Electricity & tap water connection are not essential and hence extremely useful in Rural & slum area. This technology has been transferred to several private entrepreneurs for commercialisation and deployment [6].



*Methodology of operation of
combo membrane device*



*Combo domestic water
purification device marketed
by M/S Rupali Industries*

Composite Polyamide Reverse Osmosis Membrane for Brackish Water Desalination

Reverse osmosis (RO) is an efficient water purification technology for providing safe drinking water from saline and/or contaminated water. BARC provides a process for the preparation of thin film composite polyamide (TFCP) membranes and the procedure to assemble them in commercially usable spiral modules. These brackish water RO (BWRO) membranes are capable of providing safe drinking water by removing 90% salinity from brackish water of TDS up to 5000 ppm. Besides removal of salts, it also removes/reduces virtually all the contaminants from water like turbidity, heavy metals, fluoride, arsenic, halocarbons etc. The technology can be applied for desalination of brackish water, water recovery and reuse (in pharma /biotech, waste water, electrocoating, food and beverage industries) and radioactive waste treatment. This technology has been transferred to several private entrepreneurs for commercialisation and deployment [7].



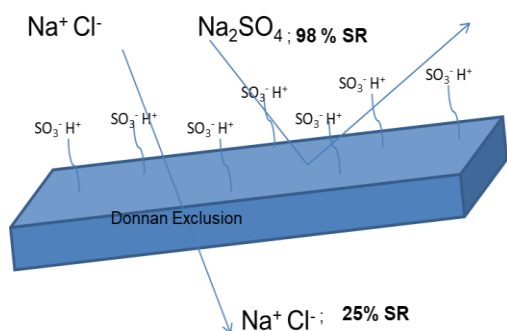
Typical TFCP - BWRO membrane



Brackish Water Reverse Osmosis Unit

Charged Nanofiltration Membranes for water treatment

Nanofiltration (NF), which operates at low pressures, offers a viable alternative to reverse osmosis that requires high energy input. The technology is based on thin film composite (TFC) type charged nanofiltration membranes. Membranes remove species containing multivalent negative charges such as sulphates and phosphates, etc. It has got applications in water purification, particularly for production of drinking water and industrial water by removal of hardness, alkalinity and partial removal of salinity from moderately brackish water.



Charged NF membrane working principle



*NF membrane modules marketed by
M/S Osmotech Membranes Pvt. Ltd.*

Hollow-fiber Ultrafiltration Membrane devices for water purifications

Ultrafiltration (UF) membranes are used to separate colloids, particulate materials, bacteria, viruses and other compounds based on principle of sieving. Hollow fibre (HF) module configuration appears promising in UF membrane applications due to its relatively higher packing density, ease of backwashing, ease of cleaning and lower pressure-drop, since it can be operated at laminar flow regime with higher cross-flow velocity. The polyethersulfone based HF ultrafiltration membrane technology were developed using in-house designed membrane spinning machine. Subsequently, technology for preparation of a prototype of HF membrane-based water purifying bottle was perfected for both ‘online’ and ‘offline’ purification of contaminated water to potable water free of turbidity and microorganisms. The bottle is capable of removing turbidity of water from > 100 NTU to less than 1 NTU and microbial removal with respect to E. coli by 4 log reduction and have purified water output of 200 ml in 5minutes in offline-mode & 900-1000ml in 2 minutes in online-mode when connected to a water tap. This technology has been transferred to several private entrepreneurs for commercialisation and deployment [8].



Fibre spinning set-up for hollow-fiber membrane preparation



HF membrane cartridge; bottle with turbid water; filtered water collected after pressurizing

ZLD Desalination System along with recovery of valuables minerals

To meet the increasing demand of fresh water, number of desalination plant has increased considerably in last decade. Currently, recovery of water from conventional desalination process is limited to 30-50%. Remaining 70-50% of the feed water is lost as concentrate, also known as 'brine'. Discharge of brine impact the aquatic environment. Management of brine in a sustainable and environment friendly way is a challenge, particularly in land locked areas. Hence, development of environmentally friendly and sustainable ZLD desalination process is the need of the hour as the most logical solution to address this problem. However, ZLD processes are capital and energy intensive, therefore cost is high.

BARC has setup pilot plant for Zero Liquid Discharge (ZLD) desalination system consisting of 10 cu.m/d brine purification unit followed with 5 cu.m/d brine concentrator unit as shown in Figure below.



10 cu.m/d Brine Purification System



5 cu.m/d Brine Concentrator Unit



In brine purification process (BPP), calcium and magnesium and sulphate ions are removed from the brine through reactive precipitation process. From the precipitated sludge, magnesium hydroxide is recovered. In the next step, brine concentrator (BC) unit, an indigenously developed falling film evaporator, is used to concentrate the brine up to 22% which is closed to its saturation level thereby minimizes the brine volume and recovers low conductivity distilled water (< 5 micro-Siemens/cm). Production of high purity sodium chloride salts (purity $> 99.9\%$) from the concentrated and purified brine has been demonstrated in lab scale and setting up a forced circulation crystallizer (FCC) unit for demonstration in pilot scale.

Concluding Remarks

Bhabha Atomic Research Centre (BARC) has developed several water purification devices and desalination technologies, as a part of its research & development efforts towards societal applications in water sector. These technologies or products are either used in standalone or hybrid mode to suit the requirement of fresh water and are backed up with robust design concepts and pilot plant studies, which can cover the needs of households, and communities. The developed water purification technologies have been demonstrated and deployed in different parts of the country for providing safe drinking water to the common mass in rural/remote areas and transferred to several private parties on non-exclusive basis. The advancements in desalination and membrane technologies developed indigenously is contributing to United Nations' Sustainable Development Goals (SDGs), including SDG 6 (Clean water and sanitation) as nation can get benefited through availability of pure drinking water by affordable, indigenous, energy efficient and eco-friendly water purification technologies.

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Metal Organic Frameworks as Novel Adsorbent Materials for the Recovery of Uranium and Palladium from Aqueous Solutions

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Summary

A simple and efficient route to develop various novel functionalized MOF materials for rapid and recovery of U(VI) and Pd(II) from aqueous medium has been demonstrated in the present study. In this connection, a set of four distinct post synthetically modified (PSM) iso-reticular metal organic frameworks were synthesized from IRMOF-3 for the recovery and sensing of U(VI) from aqueous medium. The MOFs were characterized by FTIR, Powder XRD, BET, TGA, etc. The MOFs were investigated for U(VI) sorption studies by batch technique. All the MOFs exhibit excellent sorption capacity towards U(VI) (>90%) and maximum uptake was observed at pH 6. Herein, we present synthesis of two newly functionalized MOFs, namely, UiO-66-Dithizone and UiO-66-Phenylenediamine, from UiO-66-NH₂ via post-synthetic modification (PSM). The sorption behavior studies of Pd(II) with the PSM MOFs, UiO-66-Dithizone, UiO-66-Phenylenediamine and the parent MOF UiO-66-NH₂ as a function of pH, revealed that UiO-66-Dithizone MOF showed highest sorption tendency at pH 5 followed by UiO-66-Phenylenediamine compared to parent MOF. Additionally, Density Functional Theory (DFT) calculations were also performed to gain insight into the nature of interactions operating between Pd(II) and the MOFs.

Introduction

The recovery of strategic and precious metal ions such as uranium and palladium from aqueous solutions is of significant importance in nuclear fuel cycle operations, environmental remediation, and precious metal recycling [1-5]. Conventional separation techniques often suffer from limitations such as low selectivity, secondary waste generation, and poor performance at trace concentrations. In this context, metal–organic frameworks (MOFs) have emerged as

promising next-generation adsorbent materials due to their unique structural, chemical, and surface properties. Metal–organic frameworks represent another class of advanced adsorbents composed of metal ions or clusters coordinated to organic linkers, forming highly ordered, porous crystalline structures. MOFs are distinguished by their exceptionally high surface areas, tunable pore sizes, and customizable chemical environments. These features enable selective adsorption of target metal ions through size exclusion, ion exchange, and specific coordination interactions [6-8]. Post Synthetic Modification (PSM) is the process of integrating various organic functional groups onto pristine MOFs to modify and enhance their physical and chemical properties without affecting the structural topology. PSM strategy also enables the synthesis of MOFs with enhanced structural stability. MOFs with amino pendants have harbored great attention for modification with desired organic functional groups by PSM strategies, offering different applications [8, 9].

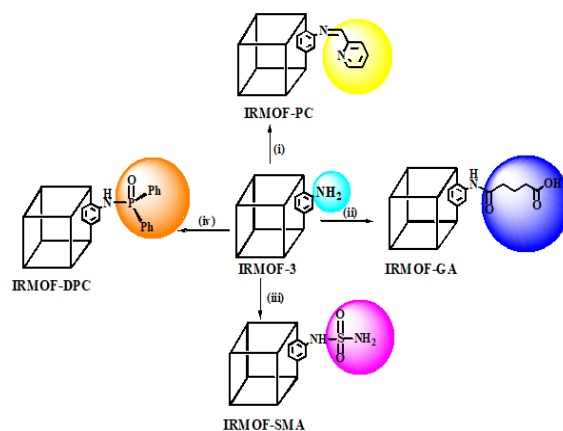
MOFs for Recovery of U(VI) from aqueous solutions

Among all energy sources, nuclear power is a dense source of power which is environmentally benign. Uranium is the widely employed fuel in nuclear industry which is environmentally toxic. Uranium resources are limited and release of uranium into ecosystems is highly undesirable. Hence monitoring and regulating uranium is crucial. Therefore, it is important to develop materials for selective sensing of U(VI) and also for its recovery from aqueous medium. For uranium recovery, MOFs containing functional groups such as $-\text{NH}_2$, $-\text{COOH}$, and phosphonate moieties have demonstrated strong binding with uranyl ions via multidentate coordination. Some MOFs also exhibit remarkable stability in acidic and radioactive environments, which is critical for nuclear fuel cycle related applications.

MOFs for Recovery of Pd(II) from aqueous solutions

The advent of widespread globalization has led to increase in consumption of platinum group metals (PGMs) due to their unique role in catalysis, pharmaceuticals, automobile industries, etc. Palladium is one of the PGMs that is in great demand and is used extensively in electronic and electrical equipment. It is also a key component of fuel cells, catalytic converters of the automotive industry, and antipollution devices. In addition, its role as a highly efficient industrial catalyst for countless chemical transformations is indispensable. Despite its high demand and

large-scale consumption, availability of palladium in earth's crust is sparse ($<10^{-6}$ %), due to which there is a need for alternative sources of palladium other than naturally occurring ores. In this context, nuclear industry provides a unique solution, where nuclear waste having a plethora of elements, can be used as an alternative source of palladium. Various porous materials like activated carbon, carbon nanotubes, graphite, graphene oxide, silica etc., have been widely employed for the recovery of palladium [11]. However, these porous materials mostly displayed lower adsorption capacity. In this context, it is important to shift focus to another class of porous materials called metal organic frameworks (MOFs) [12], which have demonstrated higher adsorption capacity. There have been various reports where MOFs have been used for the extraction of palladium from slightly acidic solutions with pH ranging from 2 to 6. In the case of palladium recovery, sulfur and nitrogen containing MOFs show enhanced selectivity toward Pd(II) ions due to soft-soft acid-base interactions, enabling efficient separation from competing metal ions [13]. MOFs offer additional advantages such as reusability, high adsorption capacities, and potential for incorporation into composite materials or fixed-bed systems. Overall, metal organic frameworks offer versatile and efficient adsorbent platform for the selective recovery of uranium and palladium from aqueous solutions, holding great promise for sustainable resource recovery and environmental protection.



Scheme 1. PSM of IRMOF-3 with various functional groups: (i) 2-pyridine carboxaldehyde; (ii) glutaric anhydride; (iii) sulfamic acid; (iv) diphenyl phosphonic chloride [14]

Synthesis and Characterization of PSM MOFs of IRMOF-3

In the last few years, iso-reticular metal organic framework-3 (IRMOF-3) has been widely used for various applications due to its high surface area (BET reports $2200 \text{ m}^2/\text{g}$) and thermal

stability ($\sim 400^\circ\text{C}$). The successful studies on PSM of amino-pendant MOFs and the properties exhibited by IRMOF-3, motivated us to perform studies on the sorption of U(VI) from the aqueous medium using functionalized IRMOFs. In this context, IRMOF-3 was synthesized by solvothermal method and PSM MOFs were prepared by incorporating different functional groups viz. 2-pyridine carboxaldehyde, glutaric anhydride, sulfamic acid, and diphenyl phosphonic chloride into IRMOF-3 (**Scheme 1**). The synthesized IRMOFs were characterized and employed for sensing and recovery of U(VI) from aqueous medium.

Different characterization techniques namely; FTIR, PXRD, TGA, NMR, SEM, and EDX were used to confirm the successful synthesis of functionalized IRMOFs. The IR spectra of PSM MOFs confirmed the presence of grafted functional groups. The PSM MOFs exhibit PXRD patterns similar to that of parent IRMOF-3 indicating the intact structure of IRMOF-3, with no apparent loss of crystallinity upon grafting of functional groups (**Figure 1**). NMR, SEM, and EDX also confirmed the formation of PSM MOFs.

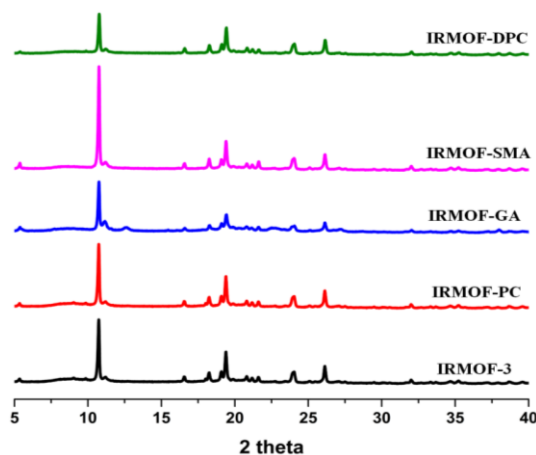


Fig. 1. Powder XRD patterns of IRMOF-3 and its functionalized MOFs

U(VI) sorption studies with IRMOF-3 and its PSM MOFs

U(VI) sorption studies from aqueous medium were carried out using IRMOF-3 and its PSM MOFs by batch method. For pH sorption studies, concentration of U(VI) (1000 mg/L), pH (2 to 9), contact time (180 min.) were optimized at room temperature ($25 \pm 1^\circ\text{C}$). About 10 mg of MOF was equilibrated with 3 mL of uranyl nitrate solution (1000 mg/L) for 3 h in an equilibration tube, at room temperature. Subsequently, the mixture was centrifuged for 10 min at 5000 rpm. The supernatant liquid was separated and used for the analysis of U(VI) by UV-

spectrophotometry using Arsenazo-III as the chromogenic agent ($\lambda_{\max} = 655 \text{ nm}$). For kinetic studies, contact time was varied from 0 to 360 min following the same procedure mentioned for pH studies. The sorption efficiency (%), distribution coefficient (K_d), and amount of uranium adsorbed (q_e) onto MOFs were calculated using the following equations:

$$\text{Sorption efficiency (\%)} = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

$$K_d = \frac{C_0 - C_e}{C_e} \times \frac{V}{m} \quad (2)$$

$$q_e = \frac{C_0 - C_e}{m} \times V \quad (3)$$

Here, C_0 and C_e denote the initial and equilibrium concentrations (mg L^{-1}) of U(VI), V is the volume of solution (L), and m is the mass of sorbent (g).

The U(VI) sorption studies performed to understand the effect of pH revealed that the sorption of U(VI) strongly depends on pH (Figure 2). Sorption of U(VI) onto IRMOFs is low up to pH 4, which increases up to pH 6 for all MOFs and then decreases after pH 7. The modified IRMOF-DPC exhibits greater sorption behaviour (300 mg/g) than that of IRMOF-3 (239 mg/g). The increasing order of sorption capacity of U(VI) onto IRMOF-3 and PSM MOFs (Table 1) are as follows: IRMOF-3-DPC (300 mg/g) > IRMOF-SMA (292 mg/g) > IRMOF-PC (289 mg/g) > IRMOF-GA (280 mg/g) > IRMOF-3 (273 mg/g). The improved sorption behaviour shown by IRMOF-DPC could be due to the strong electrostatic interaction between hard Lewis acid U(VI) and hard Lewis base: phosphoryl oxygen (P=O). The bonding efficiency of IRMOF-DPC towards U(VI) was further confirmed by IR spectroscopy. All the sorption parameters of U(VI) onto IRMOFs are presented in Table 1. In addition, desorption and recyclability studies have also been performed.

Table 1: Sorption of U(VI) onto IRMOF-3 and PSM MOFs (pH = 6)

MOF	% Sorption	K_d	q_e (mg/g)
IRMOF-3	91	3096	273
IRMOF-PC	96	8122	289
IRMOF-GA	93	4277	280
IRMOF-SMA	97	12384	292
IRMOF-DPC	99	24983	300

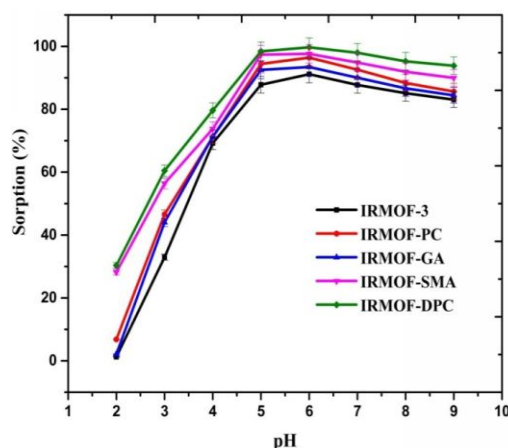
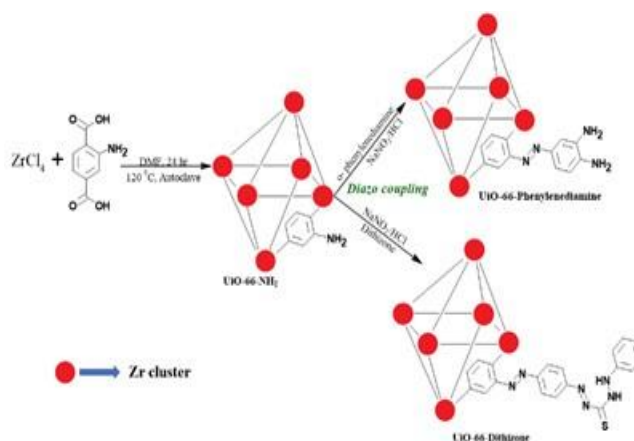


Fig. 2. Effect of solution pH on U(VI) sorption efficiency (%) onto IRMOF-3 and its PSM MOFs, $t=180$ min, $m_{\text{sorbent}}=10.0$ mg, $V_{\text{solution}}=3$ mL, $C_0=1000$ mg/L, $T=25 \pm 1^\circ\text{C}$.

Synthesis of UiO-66-NH₂ and its PSM MOFs

The extraction of palladium with the aid of MOFs shows higher adsorption capacity and kinetics as compared to other class of porous materials. Herein, we also focus on the extraction of Pd(II) from chloride medium, using zirconium based MOF, UiO-66-NH₂, which is known for its high stability owing to the presence of strong Zr(IV)-carboxylate bonds. Due to the high surface area and porosity of these class of MOFs, facile diffusion of ions/molecules occur into the three dimensional network thus enhancing the adsorption ability. Another advantage of using MOF is that appropriate functional groups can be incorporated in it, in order to tune its selectivity and adsorption efficiency. By adopting post-synthetic modification strategy, it is possible to modify the functional group in UiO-66-NH₂, to make it more selective for specific metal ions. In this



Scheme 2. Synthetic strategy for the preparation of parent and PSM MOFs

context, we have adopted PSM strategy to functionalize UiO-66-NH₂ with two different ligands such as o-phenylenediamine and dithizone, which possess soft donor atoms like N and S, thereby increasing the affinity of the functionalized MOF for Pd(II) following HSAB principle. The synthesis was carried out using the strategy given in Scheme 2 [15].

Sorption studies with Pd(II) by UiO-66-NH₂ and its PSM MOFs

The pH of solution plays a decisive role in the sorption behavior of a species on MOF. This may be ascribed to variables like variations in the metal ion's speciation with changing pH levels as well as changes in surface charge across MOF's binding sites due to protonation and deprotonation of the functional moieties.

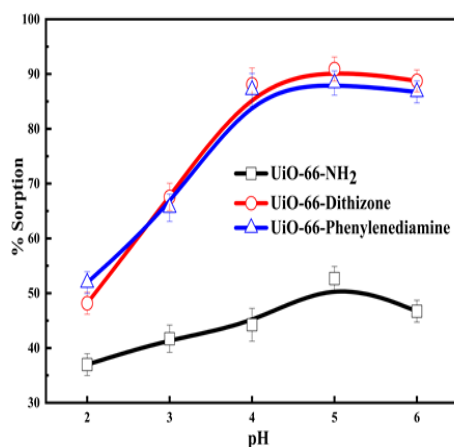
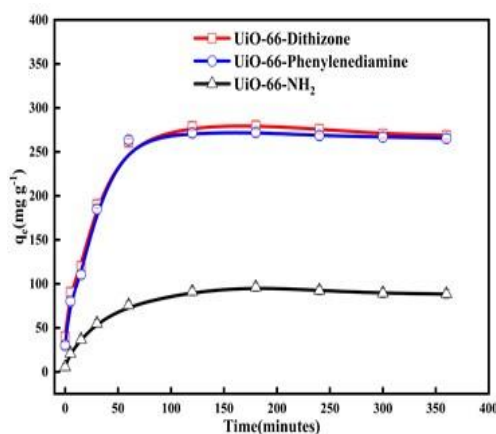


Fig. 3. The variation in sorption (%) of Pd(II) onto UiO-66-NH₂, UiO-66-Dithizone and UiO-66-Phenylenediamine with respect to pH; $m_{\text{sorbent}}=10$ mg, $t=180$ min, $V_{\text{solution}}=3$ mL, $C_0=1000$ mg L⁻¹, $T=25^\circ\text{C} \pm 1$

In Figure 3, relationship between sorption % of the parent and PSM MOFs and pH of palladium solution is explained. It was observed that with increase in pH of solution, percentage sorption increased for all three MOFs reaching a maximum at pH 5 after which decrease in sorption was observed. Percentage sorption for Pd(II) at pH 5 followed the order: UiO-66-Dithizone > UiO-66-Phenylenediamine > UiO-66-NH₂. Sorption capacity (q_e), distribution ratio (K_d) and sorption efficiency of all three MOFs at pH 5 is given in Table 2. The rate of sorption of Pd(II) on the three MOFs from aqueous solution was studied by varying the equilibration period from 0 min to 6 hours. The results revealed that there is an increase in sorption capacity till 3 hours after which it reaches almost saturation (Figure 4).

Table 2. Sorption of Pd(II) onto UiO-66-NH₂, UiO-66-Dithizone and UiO-66-Phenylenediamine at pH 5

MOF	q_e	K_d	% Sorption
UiO-66-NH ₂	96	333	52
UiO-66-Dithizone	280	3001	90
UiO-66-Phenylenediamine	272	2274	88

**Fig. 4.** The effect of contact time on the amount of Pd(II) sorbed onto UiO-66-NH₂, UiO-66-Dithizone and UiO-66-Phenylenedi amine at pH 5; $m_{\text{sorbent}}=10$ mg, $t = 0$ to 360 min, $V_{\text{solution}}= 3$ mL, $C_0= 1000$ mg L⁻¹, $T = 25^\circ\text{C}\pm 1$.

Theoretical Studies

Owing to high computational cost of modeling the entire metal-organic-framework (MOF) structure, we have adopted simplified binding ligands in our calculations that represent functional site of the MOFs. For instance, UiO-66-NH₂, UiO-66-Phenylenediamine and UiO-66-Dithizone, were modelled in our calculations as aniline, o-phenylenediamine and dithizone, respectively. These ligands and complexes with Pd(II) were subjected to systematic geometry optimization at RI-BP86-D3BJ/def2-TZVP level in order to obtain the lowest energy structures, which were further characterized as energy minima by performing harmonic frequency analysis. The lowest energy structures for the complexes are shown in Figure 5. In order to quantify complexing ability of each ligand with Pd(II), we have evaluated complexation energies for each metal complex by taking energy difference of reactants from products which shows negative values highlighting the favorable nature of the complexation process.

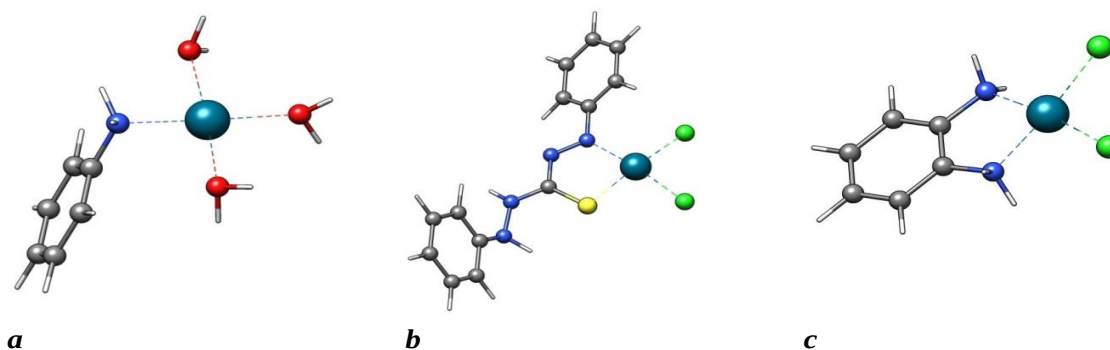


Fig. 5. Optimized geometries of the lowest energy complexes for each ligand **(a)** Pd-aniline **(b)** Pd-Dithizone **(c)** Pd-Phenylenediamine at BP86/def2-TZVP level. Color code: grey balls are carbon, blue balls are nitrogen, yellow balls are sulfur, dark cyan ball is palladium, and white colour represents hydrogen atoms.

Conclusions

PSM MOFs namely, IRMOF-PC, IRMOF-GA, IRMOF-SMA, and IRMOF-DPC were synthesised via covalent grafting of functional moieties (amide, anhydride, sulphur, and phosphorous) by modifying pendant amino (-NH_2) group present in the IRMOF-3. The resultant MOFs displayed varying porosity, surface area, stability, and binding affinities. These properties made PSM MOFs significance difference towards U(VI) sorption in aqueous medium ($\text{pH} = 2$ to 9), due to the different electrostatic interactions between grafted functional groups and U(VI). Additionally, we report synthesis of highly efficient MOFs containing nitrogen and sulfur donor groups at functional site for recovery of Pd(II) from aqueous solution. The PSM MOFs, UiO-66-Dithizone and UiO-66-Phenylenediamine showed high sorption capacity of about 280 and 272 mg g^{-1} , respectively. These values are much higher than the parent MOF, UiO-66- NH_2 showing maximum sorption capacity of 96 mg g^{-1} at $\text{pH } 5$. Mechanism of adsorption was further elucidated in DFT studies which shows adsorption of Pd(II) by means of chelation with soft N and S donor atoms in functional part of the PSM MOFs. Theoretical studies suggest that dithizone indeed has more complexing ability compared to phenylenediamine, which can be attributed to the presence of $\text{C}=\text{S}$ and N donor groups enhancing its affinity for the soft Pd(II) ions.

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About the Authors



Dr. Baliya Sreenivasulu did his M.Sc in Chemistry from University of Hyderabad and joined in Materials Chemistry & Metal Fuel Cycle Group as a Scientific Officer at IGCAR, Department of Atomic Energy. His work focuses on developing advanced solvent extraction systems and novel extractants for actinide and fission product separation in fast reactor fuel reprocessing. He combines experimental methods with computational tools like DFT to study metal–ligand interactions and has recently explored MOFs for selective actinide adsorption. With over 45 publications, he has significantly contributed to sustainable nuclear waste management. Dr. Sreenivasulu received the Young Innovator Challenge Award (FR17, IAEA, Russia) and the Young Applied Scientist/Technologist Award in 2016 by DAE.



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Modelling of Mass Transfer in Air Pulsed Columns: Comparative Study of Sieve Plate and Disc-Doughnut Column Internals

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Summary

Air pulsed columns are considered the workhorse for solvent extraction processes in the nuclear fuel cycle for offering efficient phase contact while being maintenance-free. While pulsed sieve-plate columns (PSPCs) have long been the traditional choice, the pulsed disc-and-doughnut column (PDDC) represents an innovative modification offering several advantages. In this study, the column heights required to achieve equivalent separation performance in PSPC and PDDC are compared under identical operating conditions using the axial dispersion model (ADM). Hydrodynamic variables are estimated from the earlier reported correlations and incorporated into DSXC Sim (Differential Solvent eXtraction Column Simulator), an in-house developed and rigorously validated simulator meant for prediction of the performance of differential solvent extraction contactors. The results show that the PDDC outperforms the PSPC for the desired separation task, requiring a shorter column height to attain the same degree of extraction.

Introduction

Pulsed columns serve as indispensable tools in solvent extraction processes of the nuclear fuel cycle, owing to their passive agitation mechanism driven by air pulsing. Among the various configurations of air-pulsed columns, the pulsed sieve plate column (PSPC) represents the earliest configuration featuring perforated plates (sieve plates) as internals. In contrast, the more contemporary pulsed disc and doughnut column (PDDC) employs alternately arranged disc- and doughnut-shaped baffles, offering distinct advantages (Wang, et al., 2006; Torab-Mostaedi, et al., 2011; Liu, et al., 2015). PDDC exhibits superior performance aspects, including enhanced solid-handling capacity, elimination of channeling during scale-up, and significantly higher dispersed-phase holdup. These attributes make PDDCs particularly well-suited for applications

across both the front-end (e.g., uranium refining) and back-end (e.g., spent fuel reprocessing) of the nuclear fuel cycle (Movsowitz, et al., 2000; Jahya, et al., 2009). Prior investigations from our group have provided a comprehensive comparative analysis of hydrodynamics of PSPCs and PDDCs using phase systems relevant to nuclear fuel cycle. These studies covered dispersed-phase holdup (Sarkar, et al., 2017), Sauter mean drop diameter (Sarkar, et al., 2019), flow regime transitions (Sen, et al., 2018), and axial dispersion (Sen, et al., 2021). Furthermore, empirical correlations were reported to estimate these variables accurately for both column types. The present work focuses on comparing the mass transfer in PSPCs and PDDCs through modeling based on the ADM framework. A column simulator based on ADM has been developed to predict axial concentration profiles of the solute undergoing mass transfer for both the continuous and dispersed phases in differential contactors. Hydrodynamic inputs, such as dispersed-phase holdup, drop size distribution, and axial mixing coefficients, are obtained from the correlations reported in our earlier works. A constant distribution coefficient is assumed for solute partitioning. The simulator named as Differential Solvent eXtraction Column Simulator (DSXCSim) is developed as a dedicated module of ANUSim, the nuclear chemical process and equipment simulator (Darekar, et al., 2020).

Numerical model

Axial dispersion model

The axial dispersion model (ADM) is used to predict the steady-state concentration profiles in both the aqueous and organic phases. This involves solving two coupled ADM equations simultaneously, with the interphase mass transfer term serving as the coupling source term. The steady-state formulation is adopted. The resulting concentration profiles are utilized to estimate the column height required to achieve a specified degree of separation.

The governing equations of the ADM are presented as Eq. (1) for the aqueous phase and Eq. (2) for the organic phase, respectively.

$$D_{aaq} \frac{d^2 c_{iaq}}{dz^2} - V_{aq} \frac{dc_{iaq}}{dz} - \frac{k_{iOdS}}{(1-\phi)} (c_{iorg} - c_{iorg}^*) = 0 \quad (1)$$

$$D_{aorg} \frac{d^2 c_{iorg}}{dz^2} + V_{org} \frac{dc_{iorg}}{dz} + \frac{k_{iOdS}}{\phi} (c_{iorg} - c_{iorg}^*) = 0 \quad (2)$$

Here, D_{aaq} and D_{aorg} denote the axial dispersion coefficients in the aqueous (continuous) phase and organic (dispersed) phase, respectively. V_{aq} and V_{org} represent the superficial velocities of

the aqueous and organic phases. The overall mass transfer coefficient for the i^{th} solute is designated as k_{iod} , while $S = 6\phi/d_{32}$ defines the specific interfacial area (interfacial area per unit volume of the contactor), where ϕ is the dispersed phase holdup and d_{32} is the Sauter mean drop diameter. The concentrations of the i^{th} solute in the aqueous and organic phases are denoted by c_{iaq} and c_{iorg} , respectively, with z representing the axial coordinate measured from the organic-phase inlet (bottom of the column). Hydrodynamic variables, such as axial dispersion coefficients, dispersed phase holdup, and Sauter mean diameter, are evaluated using correlations reported for both pulsed disc-and-doughnut columns (PDDC) and pulsed sieve-plate columns (PSPC) in our previous studies. Numerical solution of Eqs. (1) and (2) were implemented in an in-house developed simulator DSXCSim (GUI shown in Fig. 1).

ANUSIM:DSXCSim

Hydrodynamic Parameters	Equilibrium data	Mass transfer coefficient
Holdup [-]: 0.12	Kd_C1 [-]: 6	Kl_C1 [m/s]: 0.00001
Dax [m ² /s]: 0.0002	Kd_C2 [-]: 0.6	Kl_C2 [m/s]: 0.00001
Daxy [m ² /s]: 0.00002	Kd_C3 [-]: 1.5	Kl_C3 [m/s]: 0.00001
Sauter mean drop dia [m]: 0.002		

Column Geometry	Column Diagram	
Column Height [m]: 1.5	C1 conc. [gpl]: 200 C2 conc. [gpl]: 0 C3 conc. [gpl]: 2 Aq. in Column C1 conc. [gpl]: 0 C2 conc. [gpl]: 0 C3 conc. [gpl]: 0	C1 conc. [gpl]: 0 C2 conc. [gpl]: 0 C3 conc. [gpl]: 0 Org. out Aq. out C1 conc. [gpl]: 0 C2 conc. [gpl]: 0 C3 conc. [gpl]: 0
Column Diameter [m]: 0.038		

Operating Parameters
Continuous phase Flowrate [lph]: 30
Organic phase Flowrate [lph]: 60

Calculate

Figure 1. Screenshots of Graphical User Interface of the ADM based simulator – DSXCSim

Evaluation of hydrodynamic variables

For the pulsed disc-and-doughnut column (PDDC), the dispersed-phase holdup is calculated using the correlations given by Eq. (3) – Eq. (9). A is pulsing amplitude and f is pulsing frequency; ρ_{aq} is the density of the continuous phase; $\Delta\rho$ represents the density difference between the continuous and dispersed phases. The fractional open area of the disc/ doughnut and interfacial tension are designated by Tr and σ , respectively, while μ_{org} and μ_{aq} represent the

viscosities of the dispersed and continuous phases. The acceleration due to gravity is represented by g , and l is the characteristic length scale of the column geometry which is a function of disc spacing (h), column diameter (D) and Tr . The orifice discharge coefficient, C_0 , is assigned a standard value of 0.6. Optimum value of the constant C_Π is 0.4, C_Γ is 0.5 and C_1 is 2.8. Optimised value of the coefficients $n_1, n_2, n_3, n_4, n_5, n_6$ and n_7 are 0.65, 0.87, 3.34, -0.58, -0.08, 0, -0.1 respectively (Sarkar, et al., 2017).

$$\phi = \Pi \cdot \Phi \cdot \Psi \cdot \Gamma \quad (3)$$

$$\Pi = C_\Pi + \left[\frac{\varepsilon}{g} \left(\frac{\rho_{aq}}{g\sigma} \right)^{0.25} \right]^{n_1} \quad (4)$$

$$\Phi = \left[V_{org} \left(\frac{\rho_c}{g\sigma} \right)^{0.25} \right]^{n_2} \exp \left[n_3 V_{aq} \left(\frac{\rho_{aq}}{g\sigma} \right)^{0.25} \right] \quad (5)$$

$$\Psi = \left(\frac{\Delta\rho}{\rho_{aq}} \right)^{n_4} \left(\frac{\mu_{org}}{\mu_{aq}} \right)^{n_5} \quad (6)$$

$$\Gamma = C_\Gamma Tr^{n_6} \left[l \left(\frac{\rho_{aq}g}{\sigma} \right)^{0.5} \right]^{n_7} \quad (7)$$

$$l = \frac{h^2}{4D\sqrt{Tr}} \quad (8)$$

$$\varepsilon = \frac{4\pi^2(1-Tr^2)}{3hC_0^2Tr^2} (C_1 Af)^3 \quad (9)$$

For the PSPC, dispersed phase holdup is estimated by using Eq. (10) and Eq. (11) (Sarkar, et al., 2017) where d_h is hole diameter of the sieves.

$$\phi = 36.5 \exp[39.35|Af - (Af)_m|] V_{org}^{1.02} V_{aq}^{0.02} \Delta\rho^{-0.23} \mu_{org}^{0.52} d_h^{-0.3} Tr^{-0.4} h^{-0.4} \quad (10)$$

$$(Af)_m = 9.69 \times 10^{-3} \left(\frac{\sigma \Delta\rho^{1/4} Tr}{\mu_{org}^{3/4}} \right)^{0.33} \quad (11)$$

For the estimation of axial dispersion coefficients for PDDC, the correlations given by Eqs. (12) and (13) are used (Sen, et al., 2021). Dispersed phase axial dispersion is assumed to be 1/8 times of continuous phase axial dispersion coefficient (Sarkar, et al., 2019).

$$Sc_{aq} = 0.6 Re^{-0.885} \left(\frac{A}{D} \right)^{-0.205} \left(\frac{h}{D} \right)^{0.5}; \text{ for } \frac{Af}{V_{aq}} < 3.2 \quad (12)$$

$$Sc_{aq} = 0.000205 Re^{0.455}; \text{ for } \frac{Af}{V_{aq}} > 3.2 \quad (13)$$

Where, $Re = Afh/V_{aq}$ and $Sc_{aq} = \mu_{aq}/(\rho_{aq}D_{aaq})$, represent Reynolds and Schmidt number based on axial dispersion coefficient, respectively. For the estimation axial dispersion coefficients of the continuous phase in PSPC, the correlation given by Eqs. (14) is used (Sen, et al., 2021).

Dispersed phase axial dispersion is assumed to be 1/8 times of continuous phase axial dispersion coefficient.

$$\frac{D_{aaq}}{hV_{aq}} = 3.25 \left(\frac{\mu_c}{V_{aq} D \rho_{aq}} \right)^{0.2} \left(\frac{D}{h} \right)^{0.8} \left(\frac{Af}{V_{aq}} \right)^{0.3} Tr^{-0.8} \quad (14)$$

Sauter mean drop diameter (d_{32}) used in the calculation of specific interfacial area term used in the ADM, is estimated by using Eq. (15) for PDDC. Values of the constants and coefficient used in Eq. (15) are given in Table 1. For the PSPC Sauter mean drop diameter is estimated by using Eq. (16) (Sarkar, et al., 2019). ρ^* is reference density (of water) at 298 K (997 kg/m³). σ^* is the reference surface tension of water at 298 K (0.0728 N/m).

$$\frac{d_{32}}{\sqrt{\frac{\sigma}{\Delta \rho g}}} = K_1 Tr^{m_1} \left(\frac{h}{2} \sqrt{\frac{g \rho^*}{\sigma^*}} \right)^{m_2} \left(\frac{\mu_{aq} V_{aq}}{\sigma} \right)^{m_3} \left(\frac{V_{org}}{V_{aq}} \right)^{m_4} \left(\frac{\sigma}{\sigma^*} \right)^{m_5} \left[K_2 + \exp \left(K_3 \frac{Af}{Tr} \left(\frac{g \sigma^*}{\rho^*} \right)^{-0.25} \right) \right] \quad (15)$$

Table 1: Optimized values of the constants and coefficients of the correlation given by Eq. (15)

Plate type	K_1	K_2	K_3	m_1	m_2	m_3	m_4	m_5
PDDC	14	0.077	-3.85	0.3	0.18	0.126	0.118	0.06

$$d_{32} = 0.21 \left(\frac{\sigma}{\rho_{aq}} \right) Tr^{0.48} d_h^{0.26} h^{0.34} (Af)^{-0.8} \quad (16)$$

Overall Sherwood number is estimated from the correlation given by Eq. (17). This correlation is derived from computational fluid dynamics (CFD) simulations of mass transfer from spherical drops (Sarkar, et al., 2023). This CFD based correlation developed for estimating mass transfer coefficient has been rigorously validated for pulsed disc and doughnut columns (PDDC) across a wide range of operating conditions, encompassing both extraction and stripping in uranium processing (Sarkar, 2024). The expressions for particle Reynolds number (Re_p), Schmidt number (Sc), and overall Sherwood number (Sh) are given by Eqs. (18)–(20). Sh evaluated from Eq. (17) is used to estimate overall mass transfer coefficient by using Eq. (20). D_{aq} and D_{org} represent the diffusivity of the solute in the aqueous phase and organic phase, respectively.

$$Sh = 2 + 0.00085 Re_p^{1.13} Sc^{0.98} \quad (17)$$

$$Re_p = \frac{d_{32} (V_{aq} + V_{org} + 2Af) (\rho_{aq} \rho_{org})^{0.5}}{(\mu_{aq} \mu_{org})^{0.5}} \quad (18)$$

$$Sc = \frac{(\mu_{aq}\mu_{org})^{0.5}}{(D_{aq}D_{org})^{0.5}(\rho_{aq}\rho_{org})^{0.5}} \quad (19)$$

$$Sh = \frac{K_{Od}}{(D_{aq}D_{org})^{0.5}} \quad (20)$$

Results and Discussion

To evaluate and compare the mass transfer performance of PDDC and PSPC under the same operating conditions, a comprehensive simulation study is conducted. All simulations are performed for a column of 3-inch (76.2 mm) internal diameter with a fixed fractional open area of 23% of plates / disc and doughnut internals (Sen, et al., 2019; Jahya, et al., 2005). The geometric configurations are standardized as follows: sieve-plate hole diameter of 3 mm for PSPC (Sen, et al., 2018), disc spacing of 50 mm for PSPC, and disc spacing equal to the doughnut aperture diameter (38 mm) for PDDC (Jahya, et al., 2009). The physical properties of the dispersed phase and continuous phase are that of 30%TBP in dodecane and 3N nitric acid solution.

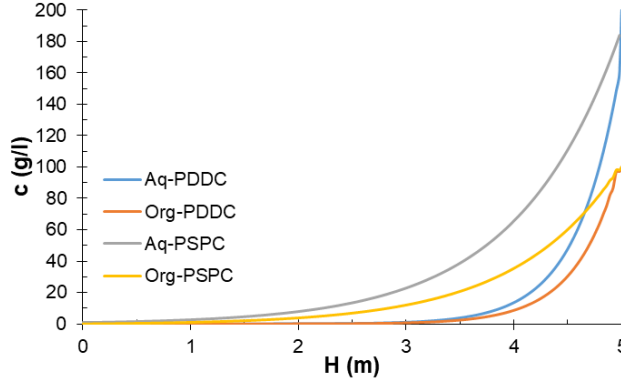


Figure 1. Axial concentration profiles of the solute in aqueous and organic phases in PDDC and PSPC for $Af = 0.025$ m/s, $V_{org}/V_{aq} = 2$, $K_d = 4$

Fig. 2 presents axial concentration profiles of the solute in the aqueous (continuous) and organic (dispersed) phases for both PDDC and PSPC in a 5 m long column. Operating conditions include an aqueous phase flow rate of 100 L/h, an organic phase flow rate of 200 L/h (phase ratio $V_{org}/V_{aq} = 2$), and a pulsing velocity of 0.025 m/s. Inlet solute concentrations are 200 g/L in the aqueous feed and 0 g/L in the organic feed, with a constant distribution coefficient (K_d) of 4, representing a typical extraction scenario. The PDDC demonstrates substantially superior

performance, achieving higher solute transfer for a given column height. This superiority is attributable to the distinctive internal geometry of the disc-and-doughnut configuration, which induces more vigorous local turbulence, greater droplet dispersion, and consequently higher dispersed phase holdup coupled with reduced Sauter mean diameters relative to the sieve-plate design.

Fig. 3 shows the column height required to achieve 99% solute transfer ($H_{99\%}$) in PDDC and PSPC for different pulsing velocities. The aqueous (continuous) phase flow rate is 100 L/h, and the organic (dispersed) phase flow rate is 200 L/h ($V_{org}/V_{aq} = 2$). Inlet solute concentrations are 200 g/L in the aqueous feed and 0 g/L in the organic feed, with a constant distribution coefficient of 4, representing a typical extraction case. As pulsing velocity increases, the required column height decreases substantially for both PDDC and PSPC. This improvement stems from reduced Sauter mean drop diameter and increased dispersed-phase holdup, which collectively increase the interfacial area per unit volume and enhance mass transfer efficiency. Across all pulsing velocities examined, PDDC consistently requires significantly less height than PSPC, underscoring its superior performance.

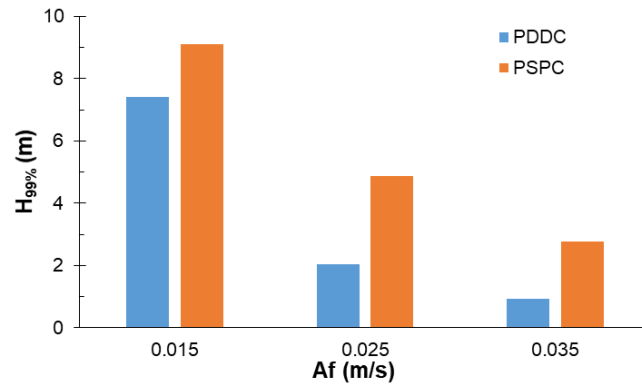


Figure 3. Height required for the 99% solute mass transfer in PDDC and PSPC for different pulsing velocities for $V_{org}/V_{aq} = 2$, $K_d = 4$

Fig. 4 compares the height required for 99% solute recovery at varying phase ratios ($V_{org}/V_{aq} = 1-3$), achieved by fixing the aqueous flow rate at 100 L/h and varying the organic flow rate from 100 to 300 L/h, while maintaining pulsing velocity at 0.025 m/s. Inlet aqueous solute concentrations are adjusted (100, 200, and 300 g/L for $O/A = 1, 2$, and 3 , respectively) to maintain equivalent solute loading. $K_d = 4$ is considered. Higher phase ratios reduce the required height in both geometries due to increased dispersed-phase holdup and consequent enhancement

of specific interfacial area. As phase ratio (V_{org}/V_{aq}) increases, driving force for the mass transfer increases resulting in enhanced mass transfer. Again, PDDC requires less height than PSPC at all phase ratios for 99% solute extraction. Interestingly, PDDC performance is more sensitive to pulsing velocity, whereas PSPC responds more strongly to phase ratio variations.

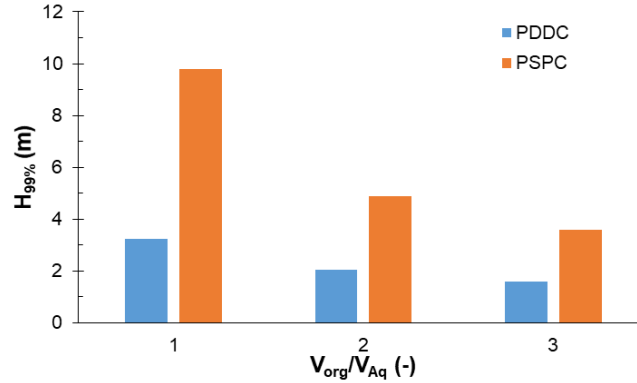


Figure 4. Height required for the 99% solute mass transfer in PDDC and PSPC for different phase ratio for $Af = 0.025$ m/s, $K_d = 4$

Fig. 5 depicts the column height required for 99% solute transfer in PDDC and PSPC for different values of distribution coefficient. The aqueous (continuous) phase flow rate is fixed at 100 L/h, the organic (dispersed) phase flow rate at 200 L/h ($V_{org}/V_{aq} = 2$), and pulsing velocity at 0.025 m/s. Inlet solute concentrations are 200 g/L in the aqueous feed and 0 g/L in the organic feed, simulating a typical extraction scenario.

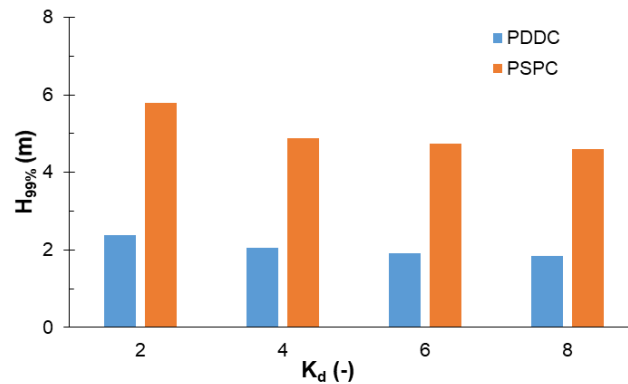


Figure 5. Height required for the 99% solute transfer in PDDC and PSPC for different distribution coefficient values for $Af = 0.025$ m/s, $V_{org}/V_{aq} = 2$

Increasing the distribution coefficient reduces the required height in both column types due to a steeper interphase concentration difference that strengthens the driving force for mass transfer. However, with increase in distribution coefficient, the height needed for 99% extraction does not decrease very significantly. The relatively modest reduction indicates that thermodynamic improvements via solvent selection exert a weaker influence on mass transfer performance compared to hydrodynamic enhancements. Consequently, superior column design provides greater efficiency gains than the selection of a more favourable solvent. At all distribution coefficient values considered, PDDC consistently requires less height than PSPC.

Conclusion

This study presents a comprehensive simulation-based comparison of mass transfer performance of PDDC and PSPC using an ADM-based framework. The model, implemented through an in house developed simulator (DSXCSim), estimates the column height required for targeted solute recovery under varied operating conditions. Hydrodynamic parameters are estimated using the correlations reported for PDDC and PSPC.

The results unequivocally demonstrate the superior performance of the PDDC over PSPC across all investigated ranges. The distinctive disc and doughnut internals generate intense local turbulence, promoting superior droplet breakup, higher dispersed-phase holdup, and smaller Sauter mean drop diameters compared to the conventional sieve plate design. These hydrodynamic advantages translate directly into enhanced specific interfacial area and markedly improved mass transfer efficiency.

Parametric analyses reveal pronounced sensitivity of column height required for 99% extraction to key operating variables such as phase ratio and pulsing intensity highlighting the critical role of operating conditions. In contrast, the influence of distribution coefficient is comparatively modest. This finding underscores a key insight that while solvent selection remains important, equipment design exerts a far stronger leverage on overall extraction performance. Superior hydrodynamic environments engineered through innovative internals deliver greater performance gains than improvements in solvent distribution coefficient.

Across all conditions examined, the PDDC consistently requires significantly shorter column heights to achieve 99% solute transfer than the PSPC. The pronounced advantages of the disc and doughnut geometry position the PDDC as a preferred contactor for demanding

extraction tasks, particularly those involving high throughput, stringent purity requirements, or spatial constraints, as encountered in the solvent extraction process of nuclear fuel cycle

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About the Authors



Dr. Sourav Sarkar obtained his B.Tech. in Chemical Engineering from the National Institute of Technology (NIT), Durgapur, in 2012, followed by an M.Tech. from the Indian Institute of Technology (IIT), Kanpur, in 2014. After graduating from the 11th batch of the Orientation Course for DGFS Fellows (OCDF) at the BARC Training School, he joined the Chemical Engineering Division of the Bhabha Atomic Research Centre (BARC). In 2024, he earned his PhD from the Homi Bhabha National Institute (HBNI). He was recipient of *Outstanding PhD Student Award* of HBNI. Dr. Sarkar is also a recipient of the *DAE Group Achievement Award*. His primary research focuses on deciphering the flow physics of chemical process equipment and solvent extraction equipment aimed at process intensification. Dr. Sarkar maintains an active scholarly presence with a portfolio of over 20 international journal publications.



Dr. K.K. Singh, PhD in Chemical Engineering from Indian Institute of Technology, Bombay joined Bhabha Atomic Research Centre in 2000 after completing one-year orientational course of BARC Training School. He conducted his postdoctoral research at TU Kaiserslautern, Germany through Alexander von Humboldt Fellowship for postdoctoral research. Presently, he is serving as Head, Process Modelling and Demonstration Section of Chemical Engineering Division, BARC. He also serves as associate professor of Homi Bhabha National Institute, Mumbai. Dr. Singh has more than 25 years of professional experience in research and development activities in the fields of computational fluid dynamics modelling of chemical process equipment in general and solvent extraction equipment in particular, multiphase flows, process intensification, chemical reaction engineering and process modelling and simulation. He is recipient of several awards. He has authored / co-authored more than 125 publications in peer-reviewed international journals.



Dr. Sulekha Mukhopadhyay, PhD in Chemical Engineering from Indian Institute of Technology, Bombay joined Bhabha Atomic Research Centre in 1991. Presently she is working as Head, Chemical Engineering Division, BARC. She is a designated professor of Engineering Science discipline of Homi Bhabha National Institute and Dean Academics of Engineering Sciences-I of BARC (CI) of HBNI. Dr. Mukhopadhyay has 35 years of professional experience in research and development, scale up and demonstration of chemical processes including synthesis of material, separation and purification techniques and process intensification. Her specific achievements include development, demonstration, on site deployment and delivery of material of importance to the department. Technology for generation of clean hydrogen by closed loop Cu-Cl thermochemical process, fluidised bed thermal denitration including direct denitration, micro bubble ozonation based technology for treatment of effluent of oil industry, bulk synthesis of CHON based Strontium selective advanced crown ether solvent for high end applications, technology for recovery of material at kg scale for departmental application, development and demonstration of intensified process and contactors for nuclear chemical separations are the salient ones besides several international publications to her credit.



Shri Kalyan Bhanja graduated in Chemical Engineering and joined BARC in the year 1990 through 34th Batch of BARC Training School. Presently, he is serving as Associate Director, Chemical Engineering Group of BARC. For the last three decades, he has been very active in the field of emerging & niche technologies, including Hydrogen Energy, specifically Electrolytic Hydrogen production, conditioning & separation, Separation & Purification technologies involving Hazardous & Flammable chemicals and Project Management involving indigenous technologies. At present he is involved in proof-of-concept demonstration, process scale-up, project planning and execution for production, separation and purification technologies.



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1	RADIOLOGICAL MEASUREMENT S	Pocket Dosimeters	Using SSD and SOP based on IAEA Report Series No. 16	1 μ Sv to 10 Sv	7.0 %
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3	RADIOLOGICAL MEASUREMENT S	Radiation Survey Instruments (Survey Meters, Area Monitors and Contamination Monitors) Pocket Dosimeters	Using SSD and SOP based on IAEA Report Series No. 16	2 mR/h to 1000 R/h	7.0 %

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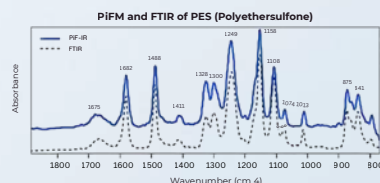
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- **PIF Laser options:** QCL (770 – 1840, 1995 – 2395 cm⁻¹), OPO/DFG (590 – 2050, 2250 – 4400, 5000 – 7000 cm⁻¹).
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ALPHA300 R – RAMAN IMAGING MICROSCOPE

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- Modular & upgradeable – scalable with AFM, SNOM, SEM integrations



ALPHA300 RS – RAMAN-SNOM MICROSCOPE

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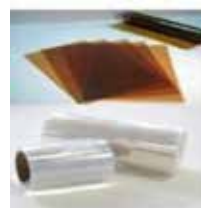
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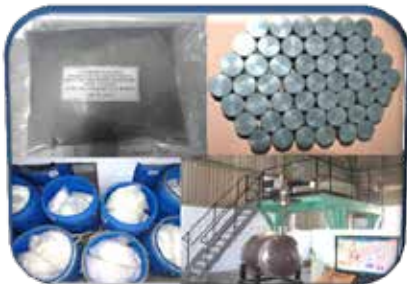
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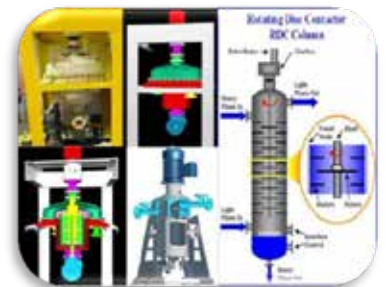
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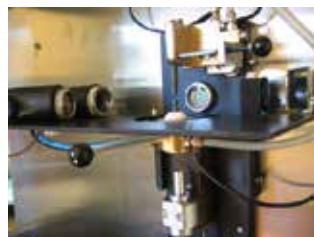
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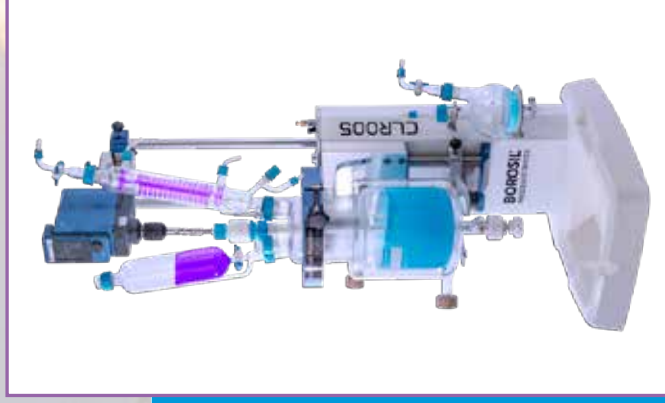
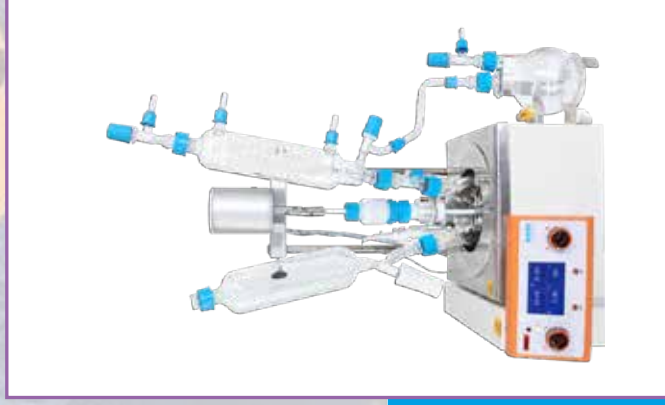
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