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DEVELOPMENT OF INNOVATIVE PROCESSES FOR THE SEPARATION OF HIGH PURITY URANIUM FROM PHOSPHORIC ACID

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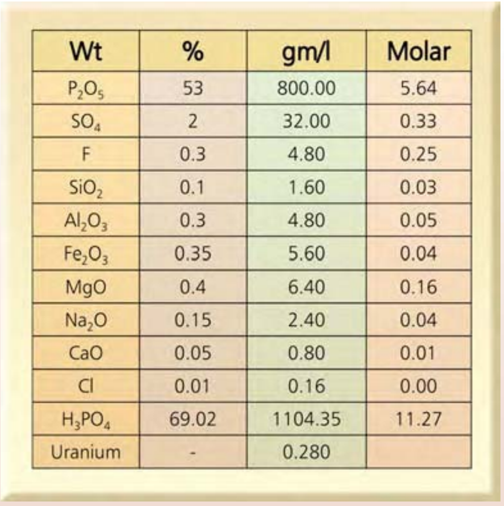
Phosphoric acid is processed in large volumes by Indian industries, into fertilizers and detergent phosphates. A major proportion of this acid contains uranium in significant concentration. Phosphates have long been recognized to be an important secondary source of uranium and indigenous development of processes of separation, has been an important area of R&D at BARC [1]. The key step in the separation flowsheet is solvent extraction. In India, the major proportion of phosphoric acid is in the concentrated form, generally called the 'Merchant Grade' Acid (MGA). The phosphate content of MGA, expressed as P_2O_5 weight percentage is ~ 55. Typical analysis is given in Table 1. Viscosity of MGA is high, in the range of 20-50 centipoise. Depending on the nature of phosphoric acid, a particular type of solvent is needed. BARC has recently developed a novel solvent extraction process based on Di-Nonyl Phenyl Phosphoric Acid (DNPPA) which has been patented [2]. Another process has been developed and patented using currently available indigenous industrial solvents for extraction from ~28% P_2O_5 weak acid (WPA) [3]. The salient details of these two processes are described in this paper, after giving a general description of the overall separation flow sheet. Some recent R&D work is summarized in the concluding part of the paper. More details are available in the published references [4-9].

Process Flow sheet for Uranium Separation from Phosphoric Acid

The overall flowsheet for separation of uranium from WPA or MGA is schematically shown in Fig. 1. It involves four types of operations, which can be described as pre-treatment, solvent extraction, 'yellow cake' recovery and post-treatment.

Table 1: Typical analysis of concentrated 'merchant grade' phosphoric acid (MGA)



Wt	%	gm/l	Molar
P_2O_5	53	800.00	5.64
SO_4	2	32.00	0.33
F	0.3	4.80	0.25
SiO_2	0.1	1.60	0.03
Al_2O_3	0.3	4.80	0.05
Fe_2O_3	0.35	5.60	0.04
MgO	0.4	6.40	0.16
Na_2O	0.15	2.40	0.04
CaO	0.05	0.80	0.01
Cl	0.01	0.16	0.00
H_3PO_4	69.02	1104.35	11.27
Uranium	-	0.280	

Pre-treatment of phosphoric acid involves cooling of the acid as a first step. The efficiency of separation is improved as a result of cooling since the subsequent solvent extraction is an exothermic equilibrium process. The phosphoric acid as produced in industries is often hot. Hence cooling to a temperature of ~30°C is carried out. A flash cooling system with high recycle ratio is preferred, since solids are present in the phosphoric acid. Solids are generated as a result of cooling, since the lowering of temperature results in supersaturation of the aqueous phase, with reference to many dissolved salts

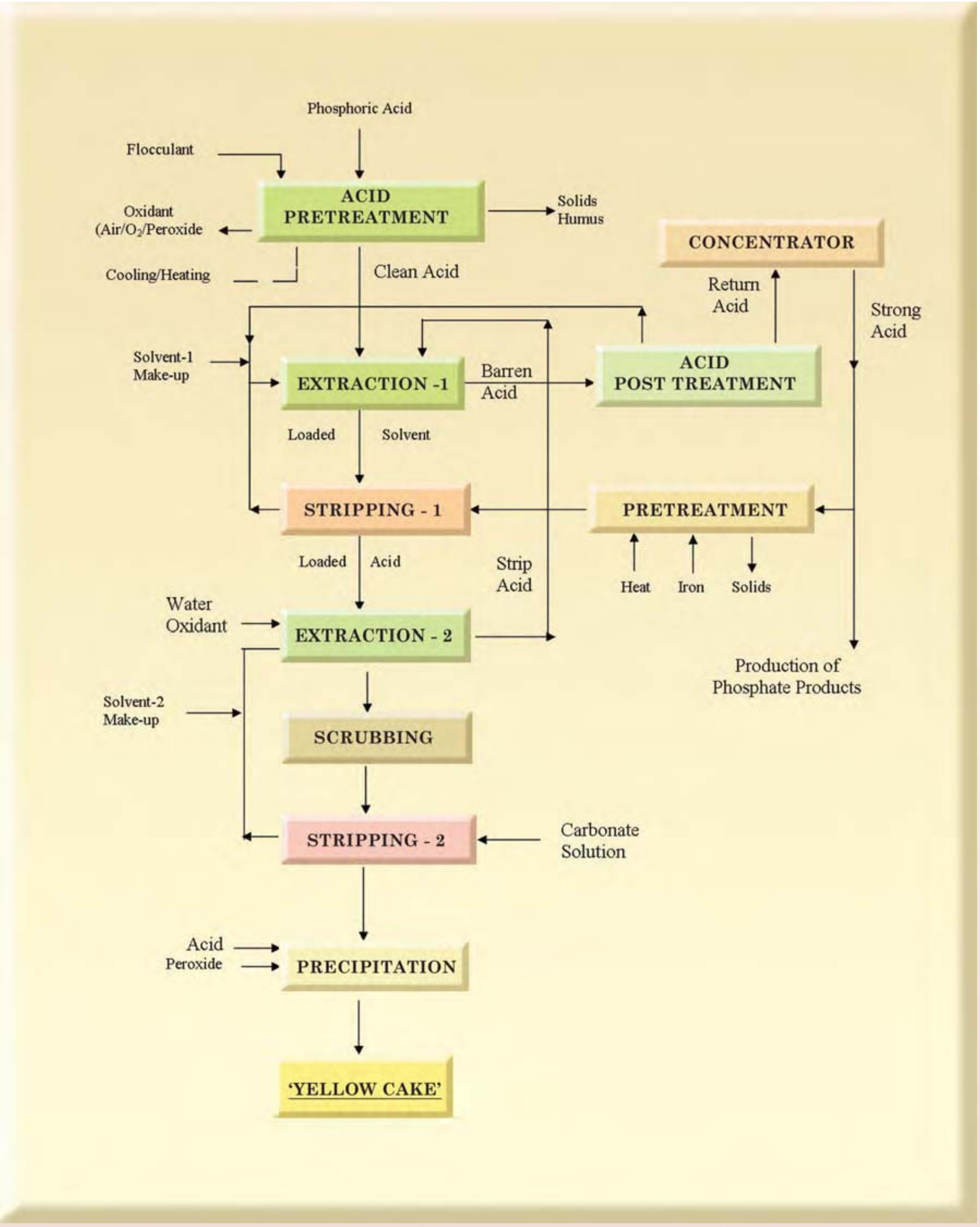


Fig. 1: Overall flowsheet for uranium separation from phosphoric acid

[10]. For smaller duties, spiral coolers can be used. The next step in pre-treatment is separation of suspended solids. This is achieved by clariflocculation with high molecular weight polymeric flocculants. Several indigenously produced flocculants have been successfully tested and product acid with <300 ppm solids was obtained, in both lab-scale and pilot-scale tests. Our studies show, that polymeric flocculants also serve to separate a significant amount of humic material from the acid. While gravity clari-flocculators are suitable for weak acid, concentrated MGA needs amplification of the gravity force in machines, such as solid bowl centrifuges. The concentrated acid is far more viscous than weaker acid and hence solid-liquid separation becomes more difficult. Separation of solids serves to ensure trouble-free down-stream operations of carbon treatment and solvent extraction. Solid separation by filtration is difficult to achieve for the viscous MGA and although it is feasible for weaker acid, it is industrially an expensive option. The third step of pre-treatment, after cooling and solid separation, is separation of the contained humic matter. Phosphoric acid produced from naturally occurring rocks, invariably contains humic matter, that can interfere with the solvent extraction. Hence, this separation is essential. It is most efficiently achieved by adsorption of humic material on activated carbon, used in the form of granules that have been packed into a series of columns. A view of such a facility is shown in Fig. 2. Pilot plant continuous test-work has been carried out, both with weak and concentrated phosphoric acids. Various grades of indigenous carbons have been tested and in particular the material prepared from coconut shells has been found to be eminently suitable. In fact, such carbons are exported from India. Tests have shown that performance of Indian carbons is superior to that of imported materials. Several cycles of column adsorption, washing and re-generation with NaOH solution have been carried out. The acid obtained from activated carbon pre-treatment, is a green acid. A cheaper option to activated carbon, involves the use of activated clays. We have evaluated a number of indigenous clays in once-through mode based on filtration. Depending on specific local conditions in a host plant, this option can be feasible. The clean green acid is subjected to the next step of pre-treatment, that involves oxidation with air or gaseous oxygen, followed by finishing with aqueous hydrogen peroxide. This step of oxidation serves to convert all the uranium from tetravalent to hexavalent state, the form which is necessary for the two innovative processes as given in the patents.

Fig. 2: Photograph of carbon columns for pre-treatment

Solvent extraction of uranium from phosphoric acid by the two processes, has several features in common, that are described herein. An important feature is the concept of a dual cycle flowsheet. In the first cycle, uranium is first extracted and then stripped into a small stream of concentrated acid. As a result of this first cycle operation, uranium concentration increases by a factor of 50-100. This cycle is optimized for high recovery with as few stages of contacting as technically needed, in order to reduce plant inventory and lower capital and operating costs. A common feature of the two patented processes, is the use of MGA as stripping agent at 55-60^{0}C under reducing conditions, achieved by the using metallic iron. Both conditions of raising the temperature and use of MGA (instead of weak acid) aid in efficiency of operation. In the second cycle of solvent extraction, the purity of uranium with respect to co-extracted impurities such as iron, rare earths and phosphate is achieved, by incorporating a set of scrubbing stages in the flowsheet. For both the patented processes, selection of a suitable scrubbing medium is a key feature of the innovation. In addition to this, the outlet scrub stream provides a potential source of by-products such as rare earths. In the second cycle, the medium used for stripping of purified uranium extract is alkaline solution of ammonium or sodium carbonate. The uranium bearing aqueous alkaline solution is further processed by precipitation of uranyl peroxide. The peroxide precipitation is a common feature of the two patented processes that yields a 'yellow cake' which is far superior to the Magnesium Diuranate (MDU) industrially produced for decades in the country. A view of samples of uranium peroxide from phosphates, diuranates and trioxide obtained from the peroxide is shown in Fig. 3.

Fig. 3: Photograph of yellow cake

The barren phosphoric acid from the solvent extraction battery is returned to the mother phosphate industry after 'post-treatment'. This involves separation of traces of solvent from the aqueous phase, with the twin objectives of: a) Solvent recovery for techno-economic operation and b) ensuring the return acid does not contain any contaminants that can interfere with the subsequent acid processing in the host industry. This step of post–treatment is carried out in a series of equipment consisting of lamellar coalescer, dissolved air floatation, counter-current diluent washing and activated carbon adsorption. The final acid is pre-heated before return, if required by the host plant. Similarly the solids that had been separated as a part of pre-treatment, are mixed with return acid, if needed by host plant, to ensure downstream operation of production of fertilizer granules is unaffected.

Next the specific innovative details of the two processes, which have been developed to meet two different indigenous requirements are described.

DNPPA-TOPO Solvent Extraction Process for uranium recovery from MGA.

This process was developed specifically in the context of indigenous industry, which uses concentrated acid (MGA) from various sources in large volumes. In fact, the largest phosphatic plants in the country use MGA as the source material rather than the weaker acid. Even plants that rely on weak acid as source material, have the option of switching over to purchased MGA in the event of non-availability of weak acid. Large volumes of the MGA handled in the industry enable economy of scale. An additional conclusion derived from the data collected by BARC is that, MGA sources are richer in uranium content, on an average, as compared to weaker acid – even when comparison is based on U/P ratio rather than concentration in ppm. The reason for this, lies in the observed higher uranium content of the rocks used for MGA, than those used for WPA.

A survey of the literature revealed that world-wide uranium recovery has been attempted only from the weaker phosphoric acid, in view of the difficulty of extraction, which is in turn related to the complexation of ionic species of uranium into less extractable form, as the acidity increases. Limited studies that have been reported [11] on the separation of uranium from concentrated acid, have involved the extraction of tetravalent uranium by a mixture of mono-ester and di-ester forms of phenyl phosphoric acid. Such processes suffer from the disadvantages that considerable amount of other impurities such as iron and rare earths are co-extracted, the stripping of uranium involves corrosive acids such as HF, the solvent system is unstable and overall recovery uneconomic. In fact, an earlier study from BARC had reported results in this direction [12] which in itself was a significant scientific advance, that phenyl phosphoric acids could be used as feasible extractants.

The inventive work covered by our patented processes, adopted a different approach, based on extraction of hexavalent uranium by a synergistic mixture of acidic organo-phosphorus compound, with a neutral synergist molecule. Such a system enables reductive stripping, adoption of dual cycle approach which yields high purity uranium, leads to minimum contamination of phosphoric acid as is desired by host fertilizer plant, avoids corrosive chemicals and has the flexibility of being integrated into a host plant, handling both weak and concentrated phosphoric acids.

In order to meet the above objective, the solvent used had to be of high purity, another point of departure from earlier work in this field, at BARC. Based on accumulated experience and literature survey, DNPPA was selected as the acidic extractant and TOPO as the synergist. Since DNPPA is not commercially available, it was synthesized by us in the laboratory. A mixture of di– and mono-esters of nonylphenyl phosphoric acid was obtained by reacting p-nonyl phenol with phosphorous oxy-chloride in the presence of pyridine in the mole ratio of 2:1:2. The reaction mixture was hydrolyzed with excess of 6 M HCl at 80^{o}C for 12 h. The di-ester and mono-ester were separated by extraction from benzene solution with 70% methanol. The unreacted nonyl phenol and neutral compounds, were separated by loading the benzene layer with neodymium (Nd) and precipitating the Nd-diester salt, in excess acetone. Di-ester was obtained by dissolving the salt in benzene and stripping with oxalic acid. The concentrations of mono-and di-esters were determined by potentiometric titration with alkali in ethanol medium. Pure DNPPA was found to be a pale yellow liquid with a density of 1.03, refractive index of 1.513, having acid number of 105.53 and molecular weight of 502.

The extraction ability of the solvent thus obtained was evaluated by contacting with synthetic phosphoric acid spiked with uranium. As shown in Table 2, the synergistic phenomenon is established by the fact, that the extraction power (measured by the distribution ratio D which is the ratio of equilibrium concentrations of uranium in the extracted organic phase to the aqueous phase) for the mixture is higher than that of DNPPA or TOPO alone. Next, the comparison of using DNPPA instead of D2EHPA which is conventional in weak acid extractions is shown in Table 3. The extraction power is increased 3-4 times when DNPPA replaces D2EHPA in the organic phase. This conclusion is based on molar comparison. Recognizing the higher molecular weight of DNPPA than that of D2EHPA, results are different when comparison is on the basis of weight. Next, the mole ratio of DNPPA to TOPO in the organic phase was varied and it was found that the optimum mole ratio is [DNPPA]/[TOPO] = 2:1 as against the optimum ratio of 4:1 for the D2EHPA – TOPO mixture. This shows that the nature of extracted species is different and there is no *a prior* rule for optimization. Next, at constant mole ratio, the DNPPA

Table 2: Extraction synergism for DNPPA-TOPO process

TOPO (M)	Mole ratio	D	
		D2EHPA	DNPPA
0.05	4:1	0.90	2.50
0.10	2:1	0.79	2.97
0.155	1.33:1	0.68	2.84

(D=Distribution ratio)

Table 3: Comparative extraction behaviour of DNPPA and D2EHPA

TOPO (M)	Mole ratio	D	
		D2EHPA	DNPPA
0.05	4:1	0.90	2.50
0.10	2:1	0.79	2.97
0.155	1.33:1	0.68	2.84

Table 4: Effect of DNPPA concentration on extraction

Sl. No.	DNPPA Conc. (M)	TOPO Conc. (M)	D
1.	0.1	0.05	0.49
2.	0.2	0.1	0.68
3.	0.4	0.2	1.08
4.	0.6	0.3	2.60

concentration was varied. Results are shown in Table 4. The increase in D value is found to follow a power law with an exponent of 0.6. It may be noted that DNPPA is a highly viscous extractant and the minimum necessary concentration in a diluent, is to be used. A further comparison of extraction of U-VI by 0.6 DNPPA + 0.3 M TOPO, with that of U-IV by 0.1 M DNPPA + 0.1 M MNPPA, showed comparable efficacy of separation, without the disadvantages associated with the extraction of tetravalent uranium, as discussed earlier.

Encouraged by the results of single-stage equilibrium extraction, tests were carried out in counter-current stages at aqueous to organic phase ratio of 1.5 and over 90% extraction of uranium was obtained in eight stages of separation. Next the reductive stripping was carried out using 12M H_3PO_4 with 10 g Fe^{2+}/L at organic to aqueous ratio of 20 and temperature of 60^0C. In five counter-current stages, with a loaded organic feed of 0.11g U_30_8/L, a pregnant aqueous solution containing > 2g U_30_8/L was obtained.

The strip solution collected in the first cycle was used as feed, after dilution and oxidation of uranium, for a second cycle. The extractant used in the second cycle was 0.3 M D2EHPA + 0.075 M TOPO. However, the phase ratio was increased in view of the dilute feed and the extract was scrubbed with dilute sulfuric acid. From the loaded organic phase, uranium was stripped with 1 M ammonium carbonate solution. The strip solution was filtered to remove traces of iron precipitate. The uranyl tri-carbonate solution contained excess ammonium carbonate and pH was found to be 8.3. Uranium precipitation was carried out using a solution of 40% H_2O_2. The neutralization was carried out with sulfuric acid. In a pH range of 3-4, the uranium precipitation was complete (>99%). The pale yellow uranium peroxide ($UO_4.xH_2O$) was filtered, washed, dried and calcined to obtain black UO_3 powder of >99% purity. Thus the entire process was proven.

An important feature of the solvent for industrial use involves stability of the solvent. In order to establish this, the solvent was contacted with feed acid for 15 days. After this period, the extraction behaviour was compared with that of freshly prepared solvent and found identical. Similarly cyclic stability tests were conducted. These involved repeated extraction and stripping. Both the composition and the phase separation behaviour were studied. It was found, that over 50 cycles could be carried out without any significant change. It was also found that low monoester content of <1% was important for stability.

The studies were also carried out using other synergistic reagents such as di-butyl butyl phosphonate (DBBP) and lower concentraction of extractants when contacting with weak phosphoric acid. In all cases satisfactory extraction with DNPPA based systems was achieved which was superior to the D2EHPA based extractants.

D2EHPA – TBP Solvent Extraction Process for Weak Phosphoric Acid

While the technical superiority of the DNPPA-TOPO process is established, its large scale industrial application is constrained by the non-availability of both the components in tonnage scale. Thus industrial exploitation of technology is presently limited to weaker phosphoric acid for which D2EHPA is adequate and BARC has proved the know how for its indigenous manufacture. But in this case difficulties were encountered in the availability of TOPO and an indigenous option was urgently needed. Hence, we developed an alternate process based on indigenously available solvents D2EHPA and TBP in a short span of less than one year.

A literature search revealed, that very limited work on the D2EHPA-TBP for uranium extraction from phosphoric acid, had been carried out. The only process reported [13] involved the use of HF for stripping and resulted in a crude uranium product that was contaminated with rare earths. Besides, there were several inconsistencies in the data. Hence, systematic parallel investigations were carried out by us incorporating innovative approaches to overcome the limitations of known processes. In particular it was found, that a suitable scrubbing stage in second cycle of two-cycle process, coupled with uranyl peroxide precipitation, yielded a product of high purity. The process was tested at several labs as well as pilot plant tests were carried out, using freshly produced phosphoric acid at industrial sites in the country. Composition of various acids is given in Table 5.

The synergism between the extraction of uranium by D2EHPA and by TBP is weaker than that of D2EHPA and TOPO. However it is significant as shown by the results given in Table 6. This significant increase translates into a very significant increase in recovery, when the effect is multiplied in a counter-current cascade. This was verified on a number of phosphoric acids and results are given in the patent.

It is shown that depending on the feed acid characteristics, over 90% uranium can be extracted in 6-8 stages operating at ~30°C using 1.5 M D2EHPA + 0.2 M TBP. The extraction is to be applied for weak phosphoric acid only, since as shown by the data given in Table 7, the extraction power sharply decreases

Table 5: Analysis of weak phosphoric acids (WPA)

Table 6: Extraction synergism for D2EHPA-TBP process

	TBP,M	D
D2EHPA 1.2 M	0	1.63
	0.05	1.86
	0.2	2.32
	0.3	1.86
	0.4	1.74
	0.5	1.54

Table 7: Effect of aqueous acidity on U extraction by D2EHPA-TBP

Acid, M	D
< 1	130
2	35.8
5	1.8
9	0.32
12	0.28
14	0.21

Table 8: Effect of aqueous acidity on yttrium extraction

H_3PO_4, M	D
3	78
5.5	2.66
6	2.02
7	0.98
8	0.17
10	0.026
12	< 0.005

with the acid concentration. The behaviour of rare earths, which are present in the phosphoric acid in concentrations often higher than uranium, is equally important. With yttrium, as a representative element, the behaviour is shown in Table 8. This data also shows that using concentrated phosphoric acid in the subsequent stripping operation, both uranium and rare earths should get transferred from the organic phase to the aqueous phase. In the first cycle of operation, this ensures that there is no build-up of these elements in the recycled organic. However in the second cycle, the separation of co-extracted uranium and rare earths into two different streams is desirable for product purity. This separation is achieved by scrubbing. Sulphuric acid is a very suitable scrubbing agent in the environment of a phosphatic fertilizer plant. Using this agent, the effect of acid concentration is shown in Table 9. Thus efficient scrubbing is feasible by adjusting the aqueous acidity.

Table 9: Effect of aqueous scrubbing acidity on Y

H_2SO_4, M	D
1.75	1.97
3.5	0.22
5.5	0.078
6.6	0.079

The precipitation of uranium after the process of dual cycle extraction, is illustrated by the experimental data described hereinafter, for a typical fertilizer acid. Five litres of the combined solution resulting from alkaline carbonate strip of second cycle, containing 22.01 g/l uranium, was mixed at room temperature of 29°C with 0.4 l of 12.5 M NaOH solution. This raised the pH to 12 and precipitated sodium diuranate. The precipitate was filtered and slurried in one litre of water and then 250 ml of 4 M sulfuric acid was added to it and the precipitate was dissolved. The insolubles were filtered out and the pH of the clear filtrate solution was adjusted to 3.5 using 80 ml of 12.5 M NaOH. After pH adjustment, 100 ml of H_2O_2 was added. This precipitated uranyl peroxide. It was filtered and the filter cake was washed with 1500 ml of acidic water. The 1300 ml filtrate and 150 ml washing analyzed 2 and 1ppm uranium respectively. The air-dried cake weighed 168 gm with uranium content of 67% by wt. With respect to uranium in the feed acid for extraction and that in the stripping acid, this represented an overall recovery of 90.7% by wt. The purity of the uranium prepared was $>$ 99.9 % by wt. The uranium peroxide filter cake, after washing was dried at 200^{o}C for 1 hr, to obtain a uranium trioxide product. Analysis of a number of samples is given in Table 10.

The stability of the D2EHPA-TBP solvent was also tested in a manner similar to that for DNPPA-TOPO. The results showed that D2EHPA-TBP is a very stable solvent under the processing conditions.

Recent advances

The R&D work in the field of uranium separation from phosphoric acid by REDS, BARC has led to industrial activity by the sister units of BARC [14] and currently two of the engineers are on project deputation. The research has led to setting up of large-scale (60m^3/h) pump-mix unit [15]. The pictorial details of a pilot scale (10m^3/h) unit at Trombay are shown in Fig. 4. Another technological innovation is the duplex mixer design as shown in Fig. 5. The process R&D has spanned to cover uranium recovery from partially neutralized and diluted acid (PNDA) [16] as well as the ‘hemi-hydrate’ (HH) acid by the 1.5 M D2EHPA + 0.375 M TOPO process[17]. The DNPPA extractant has also been studied for uranium separation from acid leach solution from ore processing [18]. An oxalate scrubbing has been tested for incorporating in the two patented processes described in this publication [19]. This variation has the potential to enable rare earths separation as a by-product of uranium from phosphoric acid. In view of the proven advantages of uranyl peroxide-derived yellow cake over magnesium diurante (MDU), which has lower purity and more importantly it leads to the ‘silica problem’ in subsequent refining. Hence the R&D work was extended to precipitation of uranium from the ion exchange product solution [20]. The peroxide precipitation process has been industrially demonstrated at UCIL for incorporation in their yellow cake production. The stability of the uranyl peroxide and effervescence during subsequent dissolution in refining process have been factors of concern. Research has shown, that by drying

Table 10: Impurity content of uranium oxide by ICP

Code	MDU	YC- 0	Yc- 1	Yc-2	Yc-3	Yc-4	Yc-5
Y	80	3.8	6.3	5.1	1.5	2.8	5.8
Gd	29	1.6	1.3	1.3	0.66	0.74	1.1
Ce	72	3.9	5.4	5.8	2.9	5.6	4.3
Dy	33	0.7	4.9	3.6	0.98	2.0	4.2
Eu	2.8	0.2	0.19	0.16	0.08	0.13	0.13
Sm	18	4.4	1.7	1.7	0.98	1.5	1.5
B	20.5	2.56	2.2				

Fig. 4: Photograph of internal details of a pump mix unit

Fig. 5: Photograph of Duplex mixer-settler

the uranyl peroxide at a suitable temperature and heating rate, stable UO_3 is obtained. This is illustrated by the data of Fig. 6. The oxide product is a very stable pure form of uranium obtained from the secondary phosphatic sources. The data on these and related aspects has been compiled [21-23]. Current areas of research in REDS, as part of Plan projects, include R&D related to dissolved and entrained solvent recovery as well as rare earths by-product recovery. Rare earth elements such as Dy_2O_3 are of interest in nuclear reactors such as AHWR while gadolinium oxide is used in PHWRs.

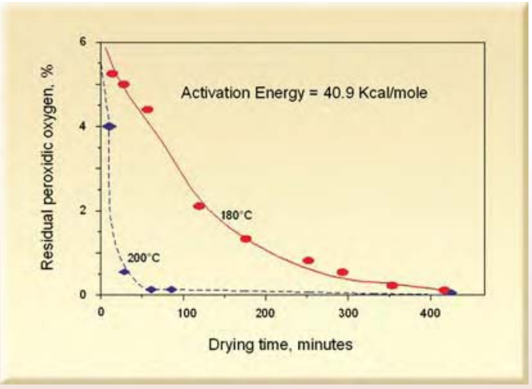


Fig. 6: Conversion of unstable uranyl peroxide to stable trioxide by drying

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MICROSPHERE IMPREGNATION TECHNIQUE FOR THE FABRICATION OF DENSE (TH,U)O_2 PELLETS FOR AHWR: A LABORATORY-SCALE STUDY

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Introduction

Thorium is an important component of fuel in India's nuclear power programme and its judicious utilization through the envisaged three stage programme is being pursued. Construction of thorium-based 300 MWe Advanced Heavy Water Reactor (AHWR) is an important step in this direction. However, one has to address some of the technical problems associated with the large scale utilization of thorium in (Th-^{233}U)O_2 MOX fuel cycle. One of these is the radiological hazard due to the presence of 500-2000 ppm of ^{232}U in the ^{233}U fuel, which demands that the entire process be carried out remotely in well shielded facilities. The main problem in fabrication of (Th, ^{233}U) MOX through conventional powder pellet route, is difficulty in automation and remotization of the mechanically intensive steps associated with the process and handling of powder generated during the process of granulation. The techniques which are more convenient and easily amenable to remote fabrication in the granulation route or the techniques that avoid the granulation route, are required, for fabrication of such fuel materials. As the time available for fuel fabrication is short after product purification of ^{233}U due to build-up of daughter products of ^{232}U, the process of purification and subsequent steps to convert the product to oxide should be simple and quick. Thus, reprocessed ^{233}U has to be used in the shortest possible time for fuel fabrication. Moreover, it is preferable to reduce the level of ^{232}U contamination of Th-^{233}U fuel to ~ 5 ppm, to get a larger time window for fabrication of Th-^{233}U fuel. The Sol-Gel Microsphere Pelletization (SGMP) is one such technique, which employs free-flowing, dust-free, sol-gel derived microspheres, as starting feed material for fabrication of the pellets. Several investigations have been done on the development of gel pelletization technique for the preparation of ThO_2 and (Th,U)O_2 pellets [1-5]. To reduce the time for fuel fabrication, many alternative routes for fabrication are being considered, which are amenable to remote handling (instead of conventional powder-pellet route). Pellet impregnation technique [6] is a suitable alternative in which a large part of the fuel fabrication step including handling of fine ThO_2 powders could be carried out, in an unshielded facility (if fresh thorium is used) and the shielded facility could be restricted to ^{233}U impregnation and sintering facilities. This process is yet to be established with respect to loading of uranium and its homogeneity across the pellet.

Microsphere impregnation technique developed by the Fuel Chemistry Division, BARC, uses porous thoria microspheres prepared by internal gelation process for impregnation of uranium. The study conducted at ORNL [7] for the preparation of (U,Pu)O_2 microspheres for pelletization showed, that gels prepared with pre-boiled HMTA-urea had enhanced crystal growth and lower tap density and crush strength. Accordingly in the present study, pre-boiled HMTA urea solution was used as gelation agent, for obtaining thoria microspheres with high porosity. Different feed compositions were used for obtaining porous thoria microspheres. Process conditions such as the concentration of uranyl nitrate solution, extent of evacuation and time of impregnation were also optimized for the preparation of (Th,U)O_2 microspheres containing 3 to 4 mole% of U, for fabrication of high density pellets.

Experimental

The process flow sheet used for the preparation of $(Th,U)O_2$ pellets in the present study is shown in Fig.1. The principal steps involved in the preparation of high density pellets are: preparation of feed solution, gelation, washing of gel spheres, heat treatment of ThO_2 microspheres, impregnation using uranyl nitrate solution under vacuum, further heat treatment, pelletization and sintering to obtain high density pellets.

Preparation of gel microspheres and characterization

Thorium nitrate solution was partially neutralized with ammonia to obtain $[NH_4^+]/\{[NH_4^+]+[Th^{4+}]\}$ ratio close to 0.5 in the final solution. The solution obtained was concentrated to yield 3 M thorium nitrate solution. The 3M HMTA-urea solution was refluxed using a water condenser for 1h and cooled to room temperature before use. The metal ion concentration ([Th]) of the feed broth was varied from 0.8 to 1.3M in the feed in steps of 0.1M. For each metal ion concentration, the [HMTA, urea]/ [Th] ratio (R) was varied in the range from 1.1 to 1.8. The gelation assembly used for the preparation of gel microspheres consists of a feed tank, gelation column, conveyor belt and wash tank. For the preparation of feed broth, thorium nitrate solution was first mixed with hexamethylenetetramine (HMTA 3M) and urea (3M) solution in the required proportion. This feed solution was then converted into droplets by forcing the solution through a 1 mm ID SS capillary. These droplets were contacted with hot silicone oil (90°C) in the glass gelation column to obtain solid gel particles. The gel particles were separated from oil, by collecting them on a conveyor belt. These particles were then washed with carbon tetrachloride to remove the adherent oil, followed by ammonium hydroxide (3M) to remove HMTA, urea, ammonium nitrate, methylol urea etc. The wet gel microspheres were characterized with respect to physical appearance and leaching behaviour during ammonia washing. The wet gel particles after ammonia washing step were dried in an air oven at 100°C for 6 h followed by heating at 250°C for 4 h. The dried microspheres

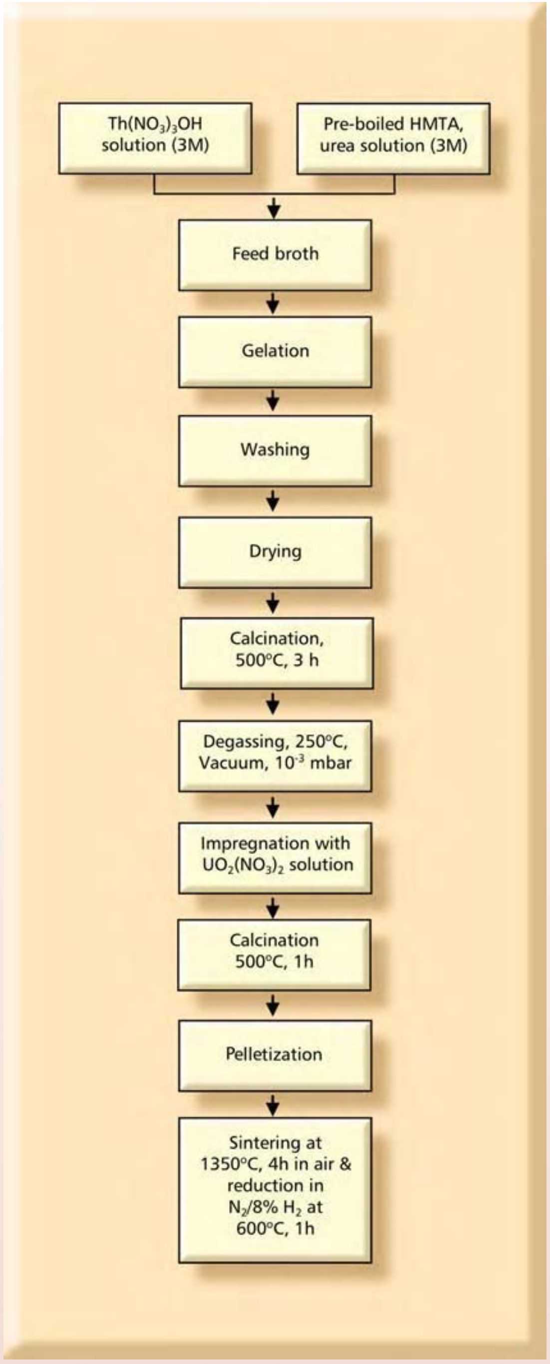


Fig. 1: Flow sheet for the preparation of $(Th,U)O_2$ pellets by microsphere impregnation technique

were further calcined at 500°C for 3 h. The dried product was characterized by measuring tap density, crush strength, specific surface area and pore size distribution.

Impregnation of uranyl nitrate solution into thoria microspheres

Impregnation was carried out using a simple indigenously designed glass apparatus (Fig. 2) consisting of a 1 litre capacity conical flask having a suitable attachment for attaining a vacuum of 10^{-2} to 10^{-3} mbar. For impregnation studies, uranyl nitrate solutions having different concentrations i.e.; 0.5M, 0.75M, 1.0M, 1.25M and 1.5M were prepared. For each impregnation study, ~ 3 g of ThO_2 microspheres were taken into the sample cell which was then inserted into the conical flask. The flask was evacuated to 10^{-3} mbar using a rotary pump. The microspheres were degassed by heating the flask at around 250°C. After degassing, the flask was isolated from the vacuum system and the spheres were cooled to room temperature. 5 to 8 ml of uranyl nitrate solution was introduced into the sample cell from the top funnel and was allowed to permeate into the thoria microspheres. The addition of uranyl nitrate solution was carried out slowly, maintaining excess of uranyl nitrate solution at the funnel, so that, the vacuum was not disturbed. The time of impregnation was varied from 5 minutes to 30 minutes. After impregnation, the microspheres were taken out from the sample cell on a wire mesh, the solution adhering to the spheres was removed by applying air jet. The microspheres were heated to 150°C followed by 500°C for 1 h. These microspheres were characterized using XRD, TG-DTA and for O/M ratio and elemental composition. The uranium content of the $(Th,U)O_2$ microspheres was determined by Davies and Grey method [8].

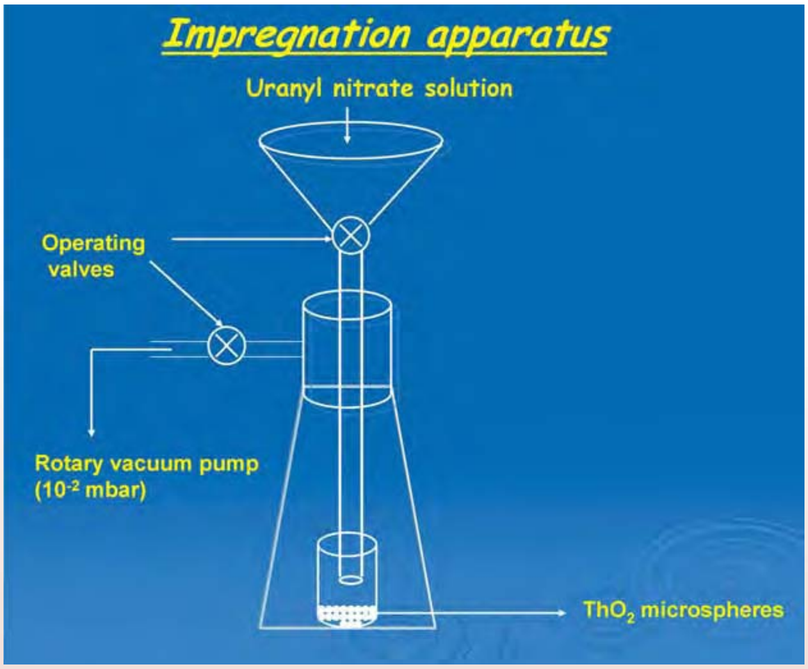


Fig. 2: Apparatus used for the impregnation of uranyl nitrate solution in ThO_2 microspheres

Results and Discussion

The wet gel microspheres were white in colour with texture varying from translucent to opaque. The translucent microspheres were hard in nature but the opaque microspheres were either hard or soft. The variation of physical characteristics of the microspheres as a function of some typical feed composition is given in Table 1.

It can be seen from Table 1 that for the preparation of microspheres using lower metal ion concentration (0.8 and 0.9 M), high R ratios (1.5 to 1.8) were required and the gels were either soft opaque or

Table 1: Variation of the characteristics of ThO_2 gel with feed composition

Sr. No.	Metal conc. [Th]	([HMTA-urea])/[Metal] (R)	Gelation time (s)	Nature of gel obtained	Leaching during ammonia washing	Nature of dried product
1	0.80*	1.50	12	Soft opaque	Yes	Cracked
2	0.80*	1.60	10	Soft opaque	Yes	Cracked
3	0.80	1.70	9	Soft opaque	Yes	50% cracked
4	0.80	1.80	9	Translucent	No	Cracked
5	0.90	1.40	22	Soft opaque	Yes	Eroded
6	0.90*	1.65	12	Translucent	No	Cracked
7	0.90*	1.70	12	Translucent	No	Cracked
8	1.15	1.3	28	Hard opaque	No	Good
9	1.15	1.35	30	Hard opaque	No	Good
10	1.20	1.35	22	Hard opaque	No	Good
11	1.25	1.15	33	Hard opaque	Slight leaching	Good

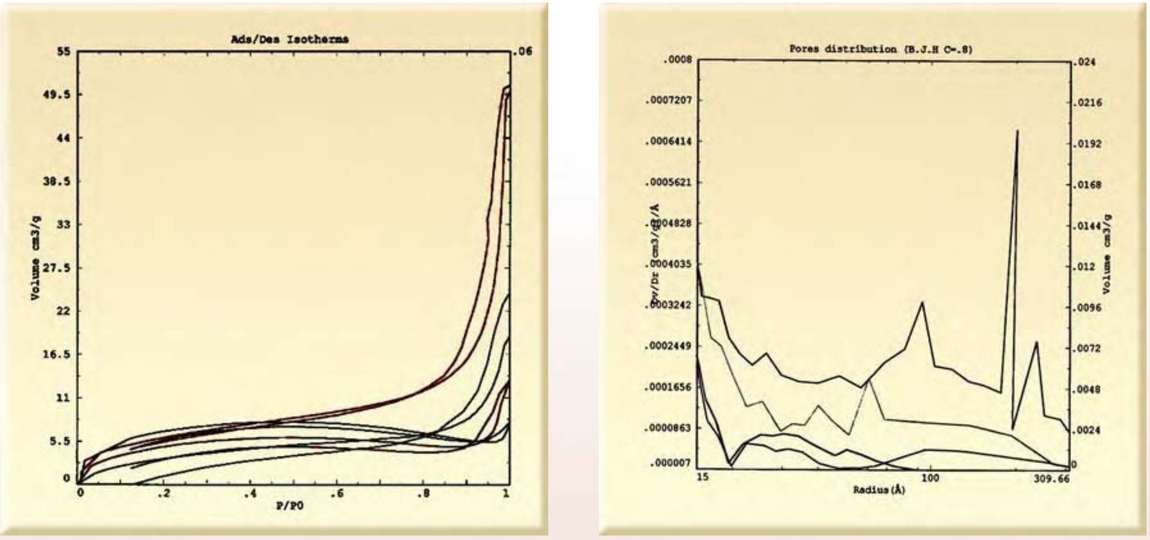
*The broth was not stable at room temperature for long

translucent in nature which cracked upon drying. Good quality microspheres, which survived the drying and calcination step were obtained with [Th] = 1.15 to1.25 and R= 1.25 to 1.35. These microspheres were used for impregnation study. The purpose of calcination at 500°C was primarily to get rid of the residual chemicals from the pores, thus exposing clean porous matrix for impregnation and to enhance the chemical stability of ThO_2 gel microspheres towards uranyl nitrate solution. The data on the variation of tap density, surface area and cumulative pore volume of heat -treated microspheres is listed in Table 2. There is a marginal increase in the tap density value (~11%) as the metal ion concentration was increased from 1.15 to 1.25 keeping R fixed at 1.35. But the specific surface area of these microspheres increased from 10.3 m^2/g to 17.7 m^2/g, which is more than 40% of the higher value.

The loading of uranium in the ThO_2 microspheres is mainly dependent on the morphology of the microspheres, i.e., the type of pores, the pore radius, their inter-connectivity and the cumulative pore volume. All compositions were found to have micro as well as meso porosity. The adsorption-desorption isotherms of 500°C heated ThO_2 microspheres as well as the pore size distribution curve is shown in Fig. 3. Considering the cumulative pore volume and pore size distribution, it can be seen that thoria microspheres prepared from feed composition [Th] = 1.25 & R = 1.15 offer the best product for impregnation.

Table 2: Variation of tap density, surface area and cumulative pore volume of ThO_2 microspheres heated at 500°C in air for 3 h

Feed Composition		Tap density (g/cc)	Specific surface area (m^2/g)	Specific pore volume (cm^3/g)	Pore radius range (Å)
[Th] M	R				
1.15	1.30	1.34	12.7	0.00497	10-300
1.15	1.35	1.29	10.3	0.00034	10-100
1.20	1.35	1.43	16.0	0.01380	10-800
1.25	1.15	1.48	21.7	0.07202	10-800
1.25	1.25	1.43	20.4	0.00890	10-100
1.25	1.35	1.45	17.7	0.0159	10-800



Adsorption-Desorption Isotherm

1 [Th]= 1.25, R= 1.15
2 [Th]= 1.20, R= 1.35
3 [Th]= 1.25, R= 1.25
4 [Th]= 1.25, R= 1.35

Pore size distribution of thoria microspheres

1 [Th]= 1.25, R= 1.15 (pore vol.= 0.072cm^3/g)
2 [Th]= 1.20, R= 1.35 (pore vol.= 0.0138cm^3/g)
3 [Th]= 1.25, R= 1.25 (pore vol.= 0.0089cm^3/g)
4 [Th]= 1.25, R= 1.35 (pore vol.= 0.0159cm^3/g)

Fig. 3: Adsorption-desorption isotherms (a) and pore size distribution (b) of ThO_2 microspheres

The adsorption-desorption isotherm pattern showed the existence of two different types of pores in the samples. According to de Boer, the hysteresis loop in the adsorption-desorption curves (the lower curve in all the hysteresis loop is of adsorption isotherm and the upper curve is of Desorption isotherm) of 1, 2 and 4 samples are of type B [9]. This type of hysteresis loop is associated with slit-shaped pores whereas that of sample 3 is of type A, which indicates the presence of cylindrical pores. To start with, most of the pores are expected to be of slit type. During calcination, the shape of the small pores is changed to cylindrical type due to the availability of larger surface energy. Even though microspheres with cylindrical pores possess high specific surface area ([Th]= 1.25, R= 1.25 from Table 2), the cumulative pore volume offered by this composition was observed to be around an order of magnitude less than that offered by microspheres, having slit type of pores. This may be due to the fact, that condensation of slit type pores is governed by smaller sides and the length of the channel may remain very large as compared to the cylindrical pores, which form by uniform condensation from all sides.

Table 3 gives the amount of uranium loaded into ThO_2 microspheres prepared with the compositions [Th] = 1.25, R = 1.15 as well as for [Th] = 1.20, R = 1.35 with varying concentrations of uranyl nitrate. Selection of these compositions for impregnation studies was based on their desirable characteristics with respect to size distribution and total open pore volume. The pore-size distribution showed, that the samples which have slit type of pores, offered more meso porosity (> 100 Å). These compositions offered more pore volumes also (Table 2). During the impregnation experiments, it was found that the maximum loading of uranium that could be achieved in the samples with cylindrical pores, was only 2 wt% even with higher concentration of uranyl nitrate solution (1.0 M). The samples with slit type pores when impregnated with uranyl nitrate, a maximum loading of 4.56 wt% of uranium could be obtained. Similarly, the loading of

Table 3: Amount of uranium loaded in ThO_2 microspheres at varying uranyl nitrate concentrations

Sample description	Molarity of uranyl nitrate	Uranium content (wt%)
[Th] = 1.25, R = 1.15	0.5M	0.34
[Th] = 1.25, R = 1.15	0.75M	1.61
[Th] = 1.25, R = 1.15	1.0M	4.56
[Th] = 1.25, R = 1.15	1.5M	4.28
[Th] = 1.2, R = 1.35	0.5M	1.89
[Th] = 1.20, R = 1.35	0.75M	3.02
[Th] = 1.20, R = 1.35	1.0M	4.35

uranium was found to be maximum with 1.0 Muranyl nitrate solution. The amount of uranium loaded using 1.5 M uranyl nitrate solution was comparable. This may be attributed to the resistance offered by the pores to the higher viscosity of 1.5 M uranyl nitrate solution.

During the impregnation experiments, when the operational vacuum exceeded 10^{-2} mbar, it was found that the microspheres crumbled, irrespective of their surface characteristics. This may be due to rapid permeation of uranyl nitrate solution into the pores. With a view to having maximum loading of uranium into microspheres without damaging the integrity of the sample, an operational vacuum of 10^{-2} mbar was optimized. Based upon the results obtained, process variables such as the operational vacuum, time of impregnation and uranyl nitrate concentrations were optimized.

It was found during pelletization, that the microspheres after impregnation, followed by calcination, become soft and can readily be compacted into good quality pellets. Pre-boiled HMTA-urea used during the preparation, renders big crystallites in the microspheres, which modify the pore characteristics favorably. Also, decomposition of uranyl nitrate solution during calcination generates strain in the gel matrix making it further soft. The green pellets prepared had a density of 4.5 to 5 g/cc. The green pellets sintered in air atmosphere at 1350°C for 4 h, prepared from microspheres containing less than 2 wt% of uranium, could not be sintered to more than 90 % of theoretical density. The pellets containing higher amount of uranium (> 3 wt %) could be sintered to ~ 96 % TD under similar conditions. This sintering temperature is much lower than the temperatures required for $(Th,U)O_2$ in reducing atmosphere [3]. It is well known that the addition of aliovalent cations such as Mg^{2+}, Nb^{5+}accelerates the sintering of thoria [10, 11]. In the present case during sintering, uranium is in higher oxidation state (>4) in air, which acts as a sintering aid. The presence of uranium in higher oxidation state in UO_{2+x} matrix is known to incorporate cationic vacancies in the matrix, which are responsible for the enhanced cation diffusivity [12]. The fractured and polished surface of the pellets showed a smooth surface without any berry structure. The microstructure of the sintered pellet viewed by SEM showed uniform size grains of 1-2 mm. Figs. 4 and 5 shows the SEM photograph of $(Th,U)O_2$ pellet sintered at 1350°C in air for 4 h.

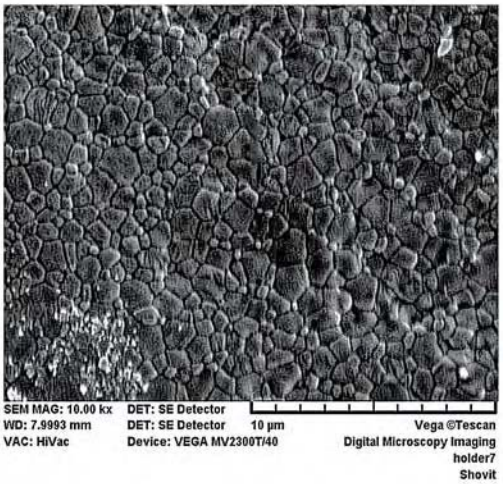


Fig. 4: SEM photograph of $(Th,U)O_2$ pellet sintered at 1350°C in air for 4 h [Th]= 1.25, R= 1.15

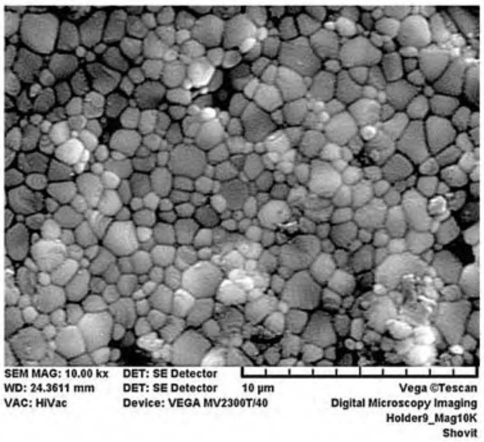


Fig. 5: SEM photograph of $(Th,U)O_2$ pellet sintered at 1350°C in air for 4 h [Th]= 1.25, R= 1.35

The elemental maps for Th and U in the microspheres as well as in the sintered pellets were recorded using Energy Dispersive X-ray to evaluate the distribution of uranium in the microspheres as well as in the final pellets. The results showed a homogeneous distribution of uranium in the microspheres as well as in the pellets for all the samples. This shows, that uranyl nitrate penetrates into the core of the microspheres rendering uniform distribution of uranium in the microspheres. Since the pellets are prepared from these microspheres, a uniform distribution of uranium across the pellets was expected. Figs. 6 and 7 give the SEM photographs of the sintered pellet as well the elemental maps which indicate the X-rays coming out of Th as well as U in the portion scanned.

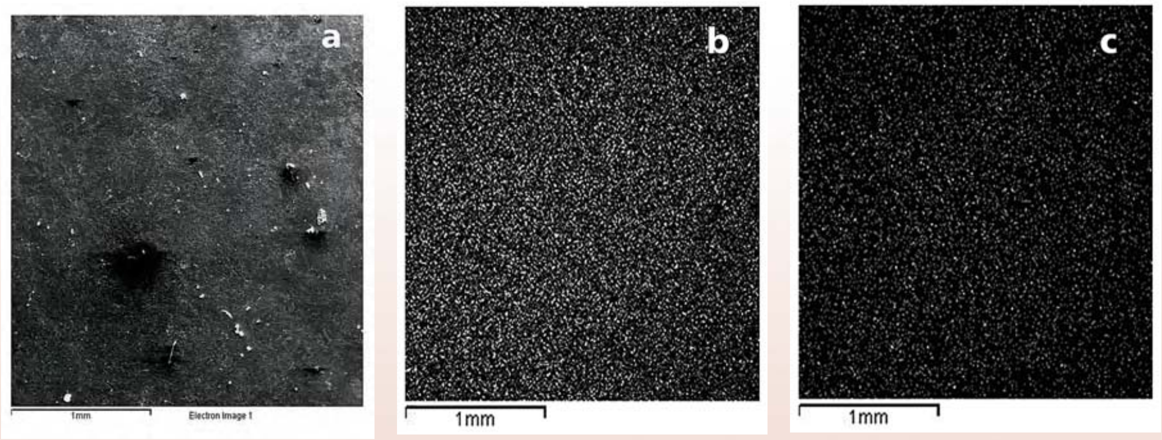


**Fig. 6: Elemental scanning photograph of pellet sample
a=portion scanned, b= x-rays from Th, c= X-rays from U**

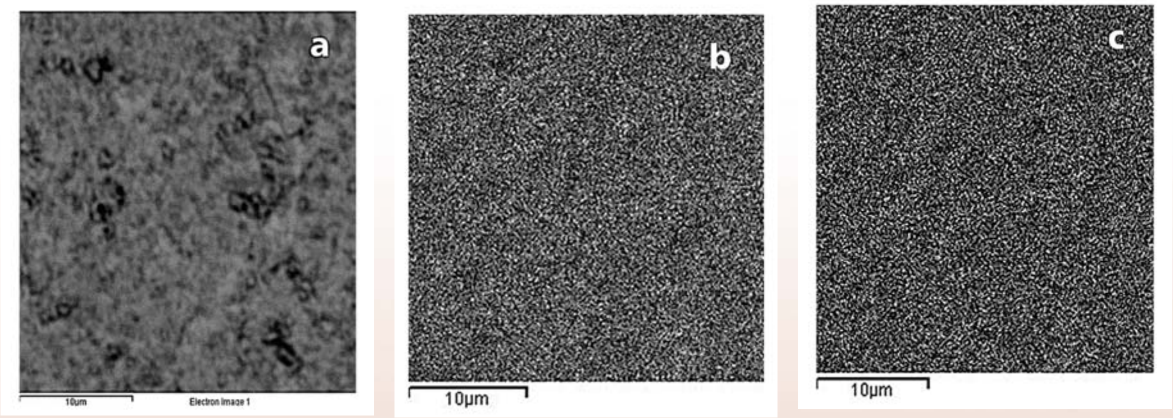


**Fig. 7: Elemental scanning photograph of microsphere sample
a= portion scanned, b= x-rays from Th, c= X-rays from U**

Conclusions

Pre-boiled HMTA-urea solution has been utilized for the preparation of soft ThO_2 microspheres having micro as well as meso porosity. Loading of uranium into the microspheres varied with operational vacuum, time of impregnation as well as the concentration of uranyl nitrate solution used for impregnation. Use of pre-boiled HMTA-urea as well as an intermediate calcination step were found responsible for obtaining microspheres which could be easily pelletized into good quality pellets. The pellets containing > 3 wt % of uranium could be sintered to ~ 96 % TD at 1350°C in air for a duration of 4 h. The elemental mapping showed that uranium is distributed in the microspheres as well as in the pellets homogeneously.

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ANNOUNCEMENT

Forthcoming Symposium
ISNS 2008

INTERNATIONAL SYMPOSIUM ON
NEUTRON SCATTERING

Under the aegis of DAE/BRNS, the above symposium would be held between Jan. 15-18, 2008, at Mumbai. The symposium would cover various aspects of neutron scattering research and applications in Physics, Chemistry, Biology and Materials Science. These would include studies of structure, dynamics and magnetism using diffraction, small-angle scattering, reflectometry and inelastic and quasi-elastic scattering, as also development of new instruments and techniques. Abstracts of papers are to be submitted by 30 th Nov. 2007 to isns2008@barc.gov.in. The deadline for registration is 30th Nov. 2007. The entire manuscript of the paper is to be submitted by 1st Jan. 2008. Further updates on the symposium would be available on http://www.barc.gov.in/symposium/isns2008.html. For any other details please contact:

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3RD ISEAC TRIENNIAL INTERNATIONAL CONFERENCE ON ELECTROANALYTICAL CHEMISTRY AND ALLIED TOPICS

(ELAC- 2007)

The third ISEAC triennial International Conference on ElectroAnalytical Chemistry and Allied Topics (ELAC-2007) was organized by the Fuel Chemistry Division, BARC under the auspices of Indian Society for ElectroAnalytical Chemistry (ISEAC). The Conference was held at Toshali Royal View Resort, Shilon Bagh, Shimla during March 10-15, 2007. After an informal social gathering of all the delegates on the evening of March 10, 2007, the Conference was inaugurated on March 11, 2007 by Dr V. Venugopal, Director, Radiochemistry and Isotope Group, BARC. During his inaugural address, he explained the role of Electrochemistry in the Nuclear Energy program of our country and formally released a bound volume containing manuscripts of invited talks and contributed papers. Dr S.K. Aggarwal, President,

Dr V. Venugopal,
Director, Radiochemistry & Isotope Group, BARC
delivering the inaugural address

Dr V. Venugopal, Director, Radiochemistry & Isotope Group, BARC releasing the bound volume containing manuscripts of invited talks and contributed papers during the inaugural function. Others standing on the dais from left; Dr Keshav Chander, Convener ELAC-2007 and Dr S.K. Aggarwal, President ISEAC, Chairman ELAC-2007 and Head, Fuel Chemistry Division.

Valedictory function: Sitting on the dais from left; Dr (Ms) J. V. Kamat, Treasurer ISEAC, Dr Keshav Chander, Convener ELAC-2007, Dr S.K. Aggarwal, President ISEAC, Chairman ELAC-2007 and Head, Fuel Chemistry Division and Mr N. Gopinath, Secretary ISEAC and Co-Convener ELAC-2007

ISEAC, Chairman, Conference Organising Committee and Head, Fuel Chemistry Division, BARC welcomed the delegates and highlighted the activities of ISEAC since its inception in October 2003. He formally released the second edition of the Directory of ISEAC Life Members. Dr Keshav Chander, Convener, Conference Organizing Committee gave a brief summary about the various topics of Electrochemistry to be covered during the Conference. Mr N. Gopinath, Secretary, ISEAC and Co-Convener, Conference Organising Committee proposed a vote of thanks.

About 100 participants including 11 overseas speakers and faculty members from Himachal Pradesh University, Shimla participated in this Conference. There were 22 Invited lectures, 27 Contributed papers (presented as posters) and 17 Research Scholars' oral papers presented during the Conference. They were grouped in to15 Technical Sessions. Invited speakers from overseas included Prof. Brian Birch (UK), Dr Shen-Ming Chen (Taiwan), Prof. Frank Endres (Germany), Dr Karel Vytras and Dr Radovan Metelka (Czech Republic), Dr Mirkin Michael (USA), Dr Jose Pingarron (France), Dr (Ms) Judith Rishpon (Israel), Dr Fwu-Shan Sheu (Singapore), Dr Woonsup Shin (South Korea) and Dr Luong John (Canada). The speakers from India included Prof. Bansi Malhotra (Delhi), Prof. P. Manisankar (Karaikudi), Dr C. Retna Raj (Kharagpur) and Dr S.K. Sarkar (BARC, Mumbai). Mr Anil Kumar from Scientific Information Resource Division (SIRD), BARC, presented a lecture on "Electrochemistry Research in India: a Scientometric View". The scientific topics covered in these invited talks included the Role of Electrochemistry in Bio-sensors, Room Temperature Ionic Liquids, Conducting polymers, Carbon nano-tubes, Screen-printed electrodes, Bismuth film electrodes etc.

During the valedictory function, some of the delegates presented their impressions about the Conference and expressed satisfaction over the high quality of technical discussions and overall arrangements during the Conference. Merit certificates and cash awards were given to the three best poster presentations and three best oral presentations. Dr S.K. Aggarwal thanked all the delegates from India and Overseas, as well as the sponsors, for their keen interest during the deliberations of the Conference. In particular, he thanked BRNS (DAE), CSIR and International Society of Electrochemistry (ISE) for co-sponsoring the Conference.

15TH NATIONAL SYMPOSIUM ON ENVIRONMENT " MITIGATION OF POLLUTANTS FOR CLEAN ENVIRONMENT": A REPORT

The Health Safety and Environment Group of the Bhabha Atomic Research Centre, Mumbai, organized the 15th National Symposium on Environment at Bharathiar University, Coimbatore during June 5 – 7, 2007 with the focal theme " Mitigation of Pollutants for Clean Environment". The symposium was organized by the Department of Physics of the Bharathiar University sponsored by BRNS, DAE. There were more than 25 invited talks from several experts across the country besides 81 contributed papers. There were 37 registered delegates from DAE and nearly 42 delegates from different universities across the country in this three-day conference. In his inaugural address, Dr S.K. Jain, Chairman and Managing Director of the Nuclear Power Corporation of India Limited and Bharathia Nabhikiya Vidyut Nigam Limited, said, that the concentrations of carbon dioxide in the atmosphere at present were higher than at any time in the past and the rate of increase of CO_2 in the atmosphere was accelerating at an alarming speed. He also appealed to the people to take measures to reduce green house gas emissions and encourage use of energy-efficient devices.

The Vice-Chancellor of Bharathiar University, Dr G. Thiruvasagam expressed concern over water pollution. Waste water from urban settlements, industries and agricultural lands were responsible for contamination of water sources. He called for proper water management and appropriate pollution control measures to mitigate pollutants. He also appealed to the government to appoint dedicated faculty to teach environmental science and to make it on par with other subjects.

In his invited talk, Mr H.S. Kushwaha, Director, Health Safety and Environment Group of BARC discussed about the importance of environmental surveillance around front end nuclear fuel cycle facilities and the dose received by the members of the public, the in-house developmental activities and future programme pertaining to environmental surveillance. In his talk Mr S.K. Chande, Vice-Chairman, Atomic Energy Regulatory Board discussed about the overview of regulatory aspects of the nuclear facilities and environment in India. In his talk Mr. R. Gupta, CMD, UCIL, Jaduguda outlined the aspects of maintaining a clean environment in the processing of uranium ore.

Dr D.T. Khathing, Registrar, North Eastern Hill University, Shillong talked about the major pollutants in hill station due to vehicular exhausts. Mr R.N. Jayaraj, Chief Executive, Nuclear Fuel Complex, Hyderabad, discussed about the operational controls for effective environmental protection followed at Nuclear Fuel Complex, Hyderabad. Dr G. Vanithakumari of Department of Zoology, Bharathiar University discussed about the environmental impact on the health of children in this millennium; a need for heightened awareness. Dr Suesh Kumar, Head, Indian Rare Earths Limited, Chavara, discussed about the environmental management for sustainable development. He also discussed about the mining and beneficiation of minerals to intermediate products generating large amount of effluents and solid wastes.

Dr L.V. Krishnan, Former Associate Director of HSEG, Kalpakkam talked about the benefits of nanotechnology and risks due to environmental mitigation. Dr A.G. Hegde of Health Physics Division, BARC discussed about the challenges in environmental radiological surveillance around Indian nuclear facilities. He also stressed upon the synergistic approach of fusing technology with analytical methods which would help to monitor the radioactive releases at just above the background and fallout levels.

In his invited talk, Mr Kanwar Raj, Head, Waste Management Division of BARC discussed about the present practices and future plans which would be followed in India, in the minimization of radioactive wastes. The application of radiation technology for the treatment of municipal sewage sludge for use in agriculture was discussed by Dr M.R. Shah of Radiation Technology Development Section of BARC. The operational experiences of MR facility, its current status and some recent developments of this technology were discussed in his presentation. Mr A.H. Khan, Raja Ramanna Fellow discussed a case study of the remediation of uranium mining legacies in eastern Germany.

Dr P. Muthusubramanian of Madurai Kamaraj University outlined the viable option for mitigation of pollutants due to the usage of solar technology. Dr S.M. Bhatia of the Bureau of Indian Standards, New Delhi discussed the standardization of measurement techniques for a clean and greener environment. He also said that implementation of standards was through legislation where specific Indian standards were referred to in the Indian government notifications wherever applicable. Dr S. Godwin Wesley of Scott Christian College, Nagarcoil, Tamil Nadu outlined the baseline study carried out for the assessment of environmental radioactivity levels around Kudankulam coast, Gulf of Munnar. His study revealed that the concentration levels and the dose rates were within limits and comparable with global level.

Dr A.R. Sundararajan of Atomic Energy Regulatory Board presented the review of Russian experiences with environmental contamination due to nuclear accidents. He also informed about the transfer coefficients of various radionuclides for different matrices in the human food chain based on this study.

In his invited talk Dr T. Mohan Kumar of Institute of Pulmonary Medicine and Research of Ramakrishna Mission Hospital, Coimbatore discussed the environmental lung diseases, their classification, generating agents etc. In his invited talk Dr S.R.Asolekar, Head, Center for Environmental Sciences and Engineering of Indian Institute of Technology, Mumbai discussed about the technologies for physico-chemical remediation of soils, aquifers and sediments. Dr P.M.B. Pillai of Health Physics Unit, Indian Rare Earths Limited, Alwaye presented the environmental Implications of solid waste containing naturally occurring radioactive materials from industrial processing of mineral and raw materials. He also projected the major issues which were associated with radiation protection and management of wastes generated by industries which process minerals and raw materials containing naturally occurring radioactive materials. In his invited talk, Mr V.D. Puranik Head, Environmental Assessment Division, BARC, outlined the sources of background radiation, people and environment.

The 82 contributed papers which were accepted for presentation were grouped into sections such as; technologies for clean environment, monitoring and modeling of pollutants and their transport, environmental radioactivity, environmental awareness, education and regulatory aspects. In the session dealing with technologies for clean environment, papers dealing with simulation studies for quantification of solid wastes during decommissioning of nuclear reactors, management of radioactive pollutants from front end nuclear fuel cycles for clean environment – a regulatory approach, *in- situ* studies on radionuclide migration at near surface waste disposal facility, development of *in-situ* pre concentration method for environmental ^{137}Cs monitoring in sea water, a protocol for the regeneration of tailings ponds at Jaduguda etc, were discussed in detail.

In the session on monitoring and modeling of pollutants and their transport, papers dealing with comparative study of dispersion characteristics of two coastal sites - KKNP and MAPS, studies of wind profile and estimation of surface layer scaling parameters for coastal sites of Tarapur; contamination transport modeling in porous media using fuzzy vertex methods; source identification of coarse and fine air particulates around Vashi; distribution of metals in the atmospheric environment of Orissa sands complex, Orissa; indoor and outdoor concentrations of carbon monoxide in Madurai city; studies of wind profile and estimation of surface layer scaling parameters for coastal site of Tarapur etc, were discussed in detail.

Participants of the 15th National Symposium on Environment (NSE-15)

In the session dealing with environmental radioactivity, papers dealing with studies on the assessment of radioactivity in vegetables and fruits grown and consumed by the residents of a typical natural high background area in India; natural radioactivity analysis of granite and stone samples from Karnataka; radiation dose due to dissolved uranium in river water; investigation of trace levels of thorium in the Mumbai harbor bay sea water; equilibrium factors in dwellings around central sector of Singhbhum thrust belt; estimation of uranium by laser fluorimeter in ambient air; estimation of environmental gamma radiation dose rates in Himalayan environment and evaluation of radionuclides in terrestrial ecosystem around proposed uranium mining site at Bagjata; outdoor thoron progeny levels in the environment of a thorium processing facility; distribution of radium-226 body burden among workers in an underground uranium mine in India; inter- comparison exercise for the estimation of low level radioactivity of man-made gamma emitters in soil samples; evaluation of a portable HPGe detector in field conditions for the measurement of terrestrial radioactivity over larger areas etc, were discussed.

In the session dealing with environmental awareness, education and regulatory aspects, papers dealing with radiological issues associated with the detection of beach sand minerals from the list of prescribed materials and its impact on marine beach ecology; proposed regulatory requirement of doses to non human species; present status and future course of action; air quality assessment in the vicinity of nuclear and thermal power plants; assessment of drinking water quality around KKNP; radioactivity content in tobacco; environmental data mining using bootstrap sampling and estimates of 95 % upper confidence; vertical profile and lythic strata of beach placer deposits of Manavalakurichi; ambient air quality in and around OSCOM due to IREL operations; preoperational environmental radiation survey around KKNPP, Tamil Nadu; Impact of chronic exposure to arsenic in combination with mercury cobalt and nickel in Cieeihnus mirgala; hydro biological features of the Bhilanganga river; a tributary of Ganga river in Garhwal Himalayas; etc were discussed in detail.

The papers presented in the poster sessions were reviewed and summarized by Dr (Ms) G.G. Pandit, Dr R.M. Tripathi of Environmental Assessment Division, BARC, Dr D.Datta of Health Physics Division, BARC and Dr P.M.B. Pillai of Health Physics Unit, IREL, Chavara.

भा.प.अ.केंद्र के वैज्ञानिकों को सम्मान
BARC SCIENTISTS HONOURED

"नंद लाल मिश्रा, संगीता धारा, खुशदेव सिंह मुढेर, उदय कुमार ठाकुर, दीप्ती शाह, आर. एम. सावंत, के.एल. रामाकुमार, एवं सुरेश के. अग्रवाल को फरवरी 14-17, 2007 के दौरान महाराजा सायाजीराव बरोडा विश्वविद्यालय, बडोदरा में आयोजित नाभिकीय एवं रेडियो रसायनिकी परिचर्चा (न्युक्लियर-2007) में" क्लोरीन डिटरमिनेशन इन (यु,पीयू)सी फ्युल बइ टोटल रिफ्लक्श्न एक्स-रे फ्लोरेसेन्स स्पेक्टक्ट्रोमीटरी "नामक शोध-पत्र की पोस्टर प्रस्तुति हेतु प्रथम पुरस्कार से सम्मानित किया। इस शोध-पत्र में यूरेनियम-प्लूटोनियम कार्बइड इंधन में क्लोरीन जैसे अधातु का अपरंपरागत विश्लेषण हेतु आने वाले टोटल रिफ्लक्शन फ्लोरिसेन्स (टीएक्सआरएफ) स्पेक्ट्रोमीटरी जैसे नवीन तकनीकों का प्रयोग किया गया है। डब्ल्यू एलए (WLa) उत्तेजित स्रोत एवं सह भीतरी मानदंड के प्रयोग से टोटल रिफ्लक्शन फ्लोरिसेन्स (टीएक्सआरएफ) स्पेक्ट्रोमीटरी के द्वारा विशलेषण करने के पश्चात यू,पीयू (U,Pu) नमूनों में क्लोरीन के चिन्ह विद्यमान थे जो कि एनएओ एच में जमा किए गए। इन परिणामों को आइ सी द्वारा मिले परिणामों से तुलना की गई। यह शोध-पत्र संगीता धारा के द्वारा प्रस्तुत किया गया।

A paper entitled "Chlorine Determination in (U,Pu)C Fuel by Total Reflection X-ray Fluorescence Spectrometry" by Nand Lal Misra, Sangita Dhara, Khush Dev Singh Mudher, Udai Kumar Thakur, Dipti Shah, R. M. Sawant, K.L. Ramakumar and Suresh K. Aggarwal, was awarded first prize (Poster presentation) Nuclear and Radiochemistry Symposium (NUCAR-2007) held at The Maharaja Sayajirao University of Baroda, Vadodara during Feb 14-17, 2007. In this paper the upcoming new technique of Total Reflection X-ray Fluorescence has been applied for a non conventional analysis of non-metals i.e. chlorine in Uranium-Plutonium carbide fuel. The chlorine present in trace levels in (U,Pu)C samples was collected in NaOH after the pyrohydrolysis of the fuel and was analyzed by Total Reflection X-ray Fluorescenec (TXRF) Spectrometry and Co internal standard. The results were compared with IC determined results. The paper was presented by Miss Sangita Dhara.

Nand Lal Misra

डॉ. नंद लाल मिश्रा ने ईंधन रसायनिकी प्रभाग में वर्ष 1983 में सदस्यता ली तथा ट्रेस अवयवों का विभिन्न सांचों में निर्धारण एवं यूरेनेटस की ठोस अवस्था, मोलिबाइट् एवं इनका ठोस समाधान एवं यूरेनिल मोलिब्डेट्स हेतू टोटल रिलेक्शन एक्सरे फ्लोरिसेन्स (टीएक्सआरएफ) के क्षेत्र में काम कर रहे हैं। मई 2000 से अप्रेल 2001 के दौरान पॉल शेरर इन्सटिट्यूट्, स्विटज़रलैंड में स्नातक की हैसियत से रहकर इन्होंने ट्रेस अवयवों का अतिशीत नमूनों हेतु टीएक्सआरएफ के अनुप्रयोग का अध्ययन किया। इन्होंने नाभिकीय पर्यावरणी एवं न्यायालयिक सांचों में धातु एवं अधातु का ट्रेस निर्धारण करने हेतु टीएक्सआरएफ विशलेषण रीति का विकास किया।

Dr Nand Lal Misra joined Fuel Chemistry Division in the year 1983 and is working in the area of application of Total Reflection X-ray Fluorescence (TXRF) for trace element determinations in different matrices and solid state chemistry of uranates, molybdates, their solid solutions and uranyl molybdates. During his stay at Paul Scherrer Institute, Switzerland from May 2000 to April 2001, as a Post Doc. Fellow, he studied the applicability of TXRF for trace element determinations in Ice Core samples. He has developed TXRF analytical methods for the trace determinations of metals and non metals in nuclear, environmental and forensic matrices.

Sangita Dhara

कुमारी संगीता धारा ने वर्ष 2004 में भाभा परमाणु अनुसंधान केंद्र के प्रशिक्षण वर्ग से ईंधन रसायनिकी प्रभाग में सदस्यता ली तथा इस समय ट्रेस अवयवों का विभिन्न सांचों में निर्धारण अल्कली धातु यूरेनेट्स तथा इनके ठोस समाधान हेतु टीएक्सआरएफ के प्रयोग के क्षेत्र में काम कर रहीं हैं। आप नाभिकीय एवं

न्यायालयिक वातावरण में टीएक्सआरएफ ट्रेस अवयवों को विभिन्न साचों निर्धारण के विकास में व्यस्त हैं।

Ms Sangita Dhara joined Fuel Chemistry Division, BARC in 2004 from the 47th batch of BARC training school and is currently working in the area of TXRF applications for trace element determinations in different matrices and solid state chemistry of alkali metal uranates and their solid solutions. She is involved in the development of TXRF methods for trace and bulk determinations of different elements in nuclear, environmental and forensic matrices.

K.D. Singh

डॉ. के.डी. सिंह मुढेर ने इंडियन इंस्टिट्यूट ऑफ टेक्नॉलोजी , मुंबई से पीएच. डी की पदवी प्राप्त करके वर्ष 1971 भाभा परमाणु अनुसंधान केंद्र की सदस्यता प्राप्त की। आप नाभिकीय पदार्थों की ठोस रसायनिकी अवस्था के विभिन्न क्षेत्रों में काम कर रहें हैं तथा एक्स-रे विवर्तन, एक्स-रे स्पेक्ट्रोमीटरी एवं उष्मीय विधियों के ठोस अवस्था के चरित्रांकण पर काम कर रहें हैं।

Dr K.D. Singh Mudher joined BARC in the year 1971 after completing his Ph.D. Degree from Indian Institute of Technology, Bombay. He has been working in the different areas of solid state chemistry of nuclear materials and has expertise in X-ray diffraction, X-ray spectrometry and Thermal methods of characterization of solid materials.

U.K. Thakur

श्री यू.के. ठाकुर ने वर्ष 2002 में भाभा परमाणु अनुसंधान केंद्र की सदस्यता प्राप्त की तथा विभिन्न नाभिकीय ईंधन में अधातु ट्रेस अवयवों का विशलेषण करने वाले वर्ग के साथ काम कर रहें हैं। आप संक्रमण के जलीय रूप प्रदीपक एवं विभिन्न आक्सीकरण अवस्थओं में एक्टिनाइड एवं धातु के क्षेत्र में कार्यरत हैं। अंतर्राष्ट्रीय पत्रिकाओं तथा विभिन्न परिचर्चाओं में 20 से भी अधिक प्रकाशन इनके श्रेय में हैं।

Mr U. K. Thakur joined B.A.R.C. in 2002 and is working with a group analyzing non-metallic trace elements in various nuclear fuels. He is working in the field of aqueous fluoride complexation of transition metal and actinides in different oxidation states. He has over 20 publications in international journals and various Symposia/Conferences his credit.

Ms D.J. Shah

श्रीमती डी.जे. शाह ने वर्ष 1990 में भाभा परमाणु अनुसंधान केंद्र, तारापुर की सदस्यता प्राप्त की। इन्होंने नाभिकीय ईंधन नमूनों में यूरेनियम एवं प्लूटोनियम के विनाशकारक तथा अविनाशकारक विशलेषण के क्षेत्र में काम किया। इन्होंने भाभा परमाणु अनुसंधान केंद्र, मुंबई में 2003 में सदस्यता ली । इस समय आप नाभिकीय उद्योग से संबधित अधातु ट्रेस अवयवों के विभिन्न सांचों पर विशलेषण करने वाले वर्ग में हैं। आप संक्रमण के जलीय रूप प्रदीपक एवं विभिन्न आक्सीकरण अवस्थओं में एक्टिनाइड एवं धातु के क्षेत्र में कार्यरत हैं।

Mrs. D. J. Shah joined B.A.R.C., Tarapur in 1990. She worked in the field of destructive and non-destructive analysis of uranium and plutonium in nuclear fuel samples. She joined B.A.R.C. Mumbai in 2003 and now she is working with a group analyzing non-metallic trace elements in various matrices relevant to nuclear industry. She worked in the field of aqueous fluoride complexing of transition metal and actinides in different oxidation states.

R.M. Sawant

डॉ. आर.एम. सावंत ने वर्ष 1980 में भाभा परमाणु अनुसंधान केंद्र की सदस्यता प्राप्त की। इन्हें वर्ष 1990 में मुंबई युनिवर्सिटी के द्वारा पीएच.डी डिग्री से सम्मानित किया गया। नाभिकीय ईंधन नमूनों में यूरेनियम एवं नाभिकीय उद्योग से संबधित अधातु ट्रेस अवयवों के विभिन्न सांचों पर विशलेषण करने में आपने महत्वपूर्ण योगदान दिया। आपने संक्रमण के जलीय रूप प्रदीपक एवं

विभिन्न आक्सीकरण अवस्थओं में एक्टिनाइड एवं धातु के क्षेत्र में विस्तृत रूप से काम किया। अंतर्राष्ट्रीय पत्रिकाओं के लिए आपने निर्णायक (रेफरी) रहें हैं डॉ. आर.एम. सावंत भाभा परमाणु अनुसंधान केंद्र के कोम्पलेक्स केमिस्ट्री वर्ग, आरएसीएस, आरसीएंडआइजी, के अध्यक्ष हैं।

Dr R. M. Sawant joined B.A.R.C. in 1980. He was awarded Ph.D. by Mumbai University in 1990. Dr Sawant has contributed significantly in developing methodologies for the analysis of non-metallic trace elements in various matrices relevant to nuclear industry. He worked extensively in the field of aqueous fluoride complexing of actinides and lanthanides in different oxidation states. He served as referee for reputed international journals. Presently, Dr R. M. Sawant is the Head, Complex Chemistry Section, RACS, RC&IG; BARC. He has to his credit two review articles and over 50 publications in international journals and various Symposia/Conferences.


K.L. Ramakumar

डॉ. के.एल.रामाकुमार ने वर्ष 1975 में भाभा परमाणु अनुसंधान केंद्र के प्रशिक्षण वर्ग के 18वें वर्ग से नाभिकीय तथा इंजीनियरिंग में ओरियंटेशन पाठ्यक्रम के पश्चात परमाणु ऊर्जा प्रभाग में सदस्यता ली। इस समय आप आरसी एवं आइजी, भाभा परमाणु अनुसंधान केंद्र के रेडियो विश्लेषी रसायनिकी अनुभाग के अध्यक्ष हैं। नाभिकीय पदार्थ की रसायनिकी गुणवत्ता नियंत्रण, मास स्पेक्ट्रोमीटरी एवं आयन - क्रोमाटोग्राफी द्वारा ट्रेस अवयवों का विभिन्न सांचों में निर्धारण, आंतरिक उष्मामापी विश्लेषण के द्वारा वाष्पीय विश्लेषण तथा नाभिकीय ईंधन का रसायनिकी विश्लेषण परीक्षण में इनकी विशेषज्ञता है। रशिया के वैज्ञानिकों को अनुदान दिलवाने हेतु डॉ. रामाकुमार अंतर्राष्ट्रीय वैज्ञानिक निर्माण (यूएसए) योजना के समकक्ष समीक्षक रहे। आप सीबसर्डोफ मे आइएईए सेफगार्डस एनेलिटिक लेबोरेटरी,आस्ट्रिया मास स्पेक्ट्रोमीटरी विश्लेषण का निरीक्षण एवं व्यासमान कार्यविधि की विशेष एवं यथार्थ स्थापना करने हेतु विशेषज्ञ की हेसियत से नियुक्ति पर गए थे। आप मुंबई विश्वविद्यालय के एक मान्यता प्राप्य गाइड हैं।

Dr K. L. Ramakumar joined the Department of Atomic Energy in 1975 after completing the 18th Batch of BARC Training School Orientation Course in Nuclear Science and Engineering. He is currently the Head, Radio Analytical Chemistry Section of RC & IG, BARC, Mumbai. He is a specialist in the fields of chemical quality control of nuclear materials, trace elemental analysis by Mass Spectrometry and Ion – chromatography, Thermal analysis by differential scanning calorimetry as well as in post - irradiation examination of nuclear fuels by chemical analysis. Dr Ramakumar has been a Peer Reviewer for International Science Foundation (USA) program for providing research grants to Russian Scientists. He was also on an expert assignment to IAEA's Safeguards Analytical Laboratory (SAL) at Seibersdorf, Austria to check and establish calibration procedures for precise and accurate mass spectrometric analysis. He is a recognized research guide of Mumbai University.


S.K. Aggarwal

डॉ. एस.के.अग्रवाल ने भाभा परमाणु अनुसंधान केंद्र के प्रशिक्षण वर्ग के 16वें वर्ग से स्नातीकरण करके 1973 भाभा परमाणु अनुसंधान केंद्र में सदस्यता प्राप्त की। इन्होंने मुंबई विश्वविद्यालय में वर्ष 1980 में पीएच.डी की उपाधि प्राप्त करके वर्ष 1987-89 के दौरान वर्जीनिया विश्वविद्यालय, हेल्थ साइन्सिस सेंटर, वर्जीनिया यूएसए में पोस्ट स्नातक की हेसियत से कार्यारंभ किया। अवयव विश्लेष्नात्मक तकनीक , मास स्पेक्ट्रोमीटरी , ए- स्पेक्ट्रोमीटरी, इलेक्ट्रो विश्लेष्नात्मक तकनीक, एवं टीएक्सआरएफ विशकर नाभिकीय पदार्थों में इनकी विशेषज्ञता है। डॉ अग्रवाल मास स्पेक्ट्रोमीटरी में विशेषज्ञ की हेसियत से कई देशों में निरीक्षण के लिए रह चुकें हैं। इस समय आप ईंधन रसायनिकी प्रभाग के अध्यक्ष हैं ।

Dr S.K. Aggarwal joined Bhabha Atomic Research Centre in 1973 after graduating from 16th batch of BARC Training School. He did his PhD from Mumbai University in 1980 and worked as Post Doctoral Fellow at University of Virginia Health Sciences Centre, Virginia, USA during 1987-89. He has expertise in elemental analytical techniques e.g. Mass Spectrometry, r-spectrometry, Electro analytical techniques and TXRF especially for nuclear materials. Dr Aggarwal has visited several foreign countries as an expert in Mass Spectrometry. Currently, he is Head of Fuel Chemistry Division.

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