

## Study of acoustic properties of nuclear extractants and its application

R Giri & G Nath\*

Department of Physics, Veer Surendra Sai University of Technology, Sambalpur 768 018, India

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In nuclear industries and different reprocessing centre, for radionuclide production, reprocessing of the nuclear reactor material and extraction of rare earth materials, solvent extraction is a significant technique. Easily available and cost effective liquid like tri butyl phosphate (TBP) (extractant) and toluene (diluent) have been used in this study for extraction cerium from rare earth material like cerium oxide ( $\text{CeO}_2$ ). The application sound theory derived from the propagation of ultrasonic wave in the treatment medium has been applied to investigate the different physico-chemical properties of mixture of extractant and diluent. Different sound wave parameters have been demonstrated using the experimentally calculated value of ultrasonic velocity, density and viscosity. Molecular interactions present inside the binary mixture are the main basis to study the variations of physical and acoustical parameters. After investigating all the parameters in terms of molecular interaction an optimum value for extraction has been established. Cerium has been separated from  $\text{CeO}_2$  by phase separation using the biphasic system and maximum extraction efficiency has been examined using Nernst equation.

**Keywords:** Solvent extraction, Tri butyl phosphate, Toluene, Ultrasonic velocity, Acoustic parameter

### 1 Introduction

Extraction and reprocessing of spent nuclear fuels in an easier way are challenging topic in the field of nuclear fuel cycle. There are different conventional methods already applied in this area having some difficulties for reprocessing of the nuclear fuels. It is necessary to investigate convenient and cost effective method in this regard to get maximum product. Solvent extraction technique is one of the best ways for extraction and separation process of rare earth elements as well as nuclear wastes. Different organic solvents are used by many people for extraction purpose resulting in high corrosion to the apparatus, but application of large amount of such type of toxic and volatile organic solvents are hazardous for both human being as well as environment. For this reason addition of diluent is a wise way for improvement of extraction efficiency without any problem to the apparatus. So the actual function of the diluent inside the extractant is not still well explained even if many works have already done in this field<sup>1-6</sup>. By studying mutual interactions between extractant-diluent pair, to find out the optimum concentration for extraction is severe concern for all the researchers working in this area. So our main aim of the current analysis is to demonstrate the optimum concentration range of EDP for extraction of rare earth materials by increasing the effectiveness of extraction.

Ultrasonic technique is the best way to understand the basic mechanism present inside the solvent by using some acoustical parameters. It is a non destructive technique which can easily applicable to the liquid mixture without doing any harm to the fundamental behavior of the sample. TBP is a viscous liquid that can be compatible with the non polar liquid like toluene for performing extraction process. TBP has been widely used as an extractant in different industries for extraction and reprocessing of the rare earth materials. It is an easily available chemical with outstanding chemical resistance and suitable physical properties. Due to presence of phosphoryl group ( $\text{P}=\text{O}$ ) in TBP forming adducts or solvates with the metal ions, gives significant results in the field separation. Thus, it is necessary to analyze different physico-chemical properties of TBP-toluene mixture. In applications of liquid-liquid extraction processes for recycling of used nuclear fuel, the aqueous phase co-exists with an organic extracting phase which consists of a mixture of an extracting agent and a diluent. Cerium is a member of the lanthanide series of metals and is the most abundant of the rare-earth elements in the earth's crust. Cerium exists in both the trivalent ( $\text{Ce}^{3+}$ ) and the tetravalent ( $\text{Ce}^{4+}$ ) state. Cerium is found in nature along with other lanthanide elements in the minerals like alanite, bastanite, monazite, cerite, and samarskite; however, only bastanite and monazite are important sources commercially. Because of its unique stability in the

\*Corresponding author (E-mail: ganesh\_nath99@yahoo.co.in)

tetravalent state cerium can be separated out from the other rare-earth elements through oxidation (forming  $\text{CeO}_2$ ) followed by variable solubility filtration.

## 2 Materials and Instruments

Highly pure and analytical grade chemicals like tributyl phosphate (TBP) (AR > 98%), toluene (AR > 99%), nitric acid ( $\text{HNO}_3$ ) (AR > 15.5 mol/L) and Cerium Oxide  $\text{CeO}_2$  (AR > 99%) were acquired from CDH Chemicals Pvt. Ltd. The samples were used as received. The solvent mixture were prepared on percentage basis (w/w) by mixing known mass of toluene in appropriate masses of TBP and measuring their masses with the help of a high precision electronic balance of (WENSAR, PGB 100, with accuracy  $\pm 0.001$  gm). The densities of all pure liquids and the mixtures were measured by a specific gravity bottle calibrated with deionised double distilled water of density  $0.9960 \times 10^3 \text{ kg/m}^3$  for different temperature ranges. The precision of density measurement was within  $\pm 0.0001 \text{ kg/m}^3$ . The sound velocities of pure liquid as well as the mixtures were evaluated at various temperatures (precise up to  $\pm 0.01$  m/s) by a single crystal variable path multi frequency ultrasonic interferometer operating at different frequencies 1 MHz-4 MHz (Mittal Enterprises, New Delhi, Model-M-81S). The temperature of the mixture was maintained constant within  $\pm 0.01$  K by water

circulating bath (B-206). Viscosities of the prepared sample were measured by Redwood apparatus MAC, #RWV-5271 precise up to  $\pm 0.0001 \text{ Ns.m}^{-2}$ . To perform the extraction process of cerium, a magnetic stirrer (MAC, Model-#MSHP-2) and an ultrasonic bath (EMCOLITE-Ultrasonics, 125 MHz) were used to equilibrate both organic and aqueous phase.

## 3 Experimental Procedure

Different mole percentage of solvent mixture was prepared by adding TBP with toluene. Prepared samples were preserved in ground-glass stopper bottles to avoid environmental contamination. Ultrasonic technique was applied to examine the concentrations of extractant-diluent in terms of existence of different molecular interaction. Density and viscosity of the pure liquid as well as the mixtures was measured. Using ultrasonic interferometer ultrasonic velocity of solvent mixtures of TBP with toluene was measured for different frequencies like 1 MHz - 4 MHz and for different temperature such as 303.15 K, 308.15 K and 313.15 K. The basic sound wave theory was used to determine the velocity of the liquid mixture. The temperature of the solution was controlled to a desired value by water circulating bath as shown in Fig. 1. Equal volumes of two phases, organic and aqueous phases were equilibrated for extraction in a separatory funnel. Organic phase was

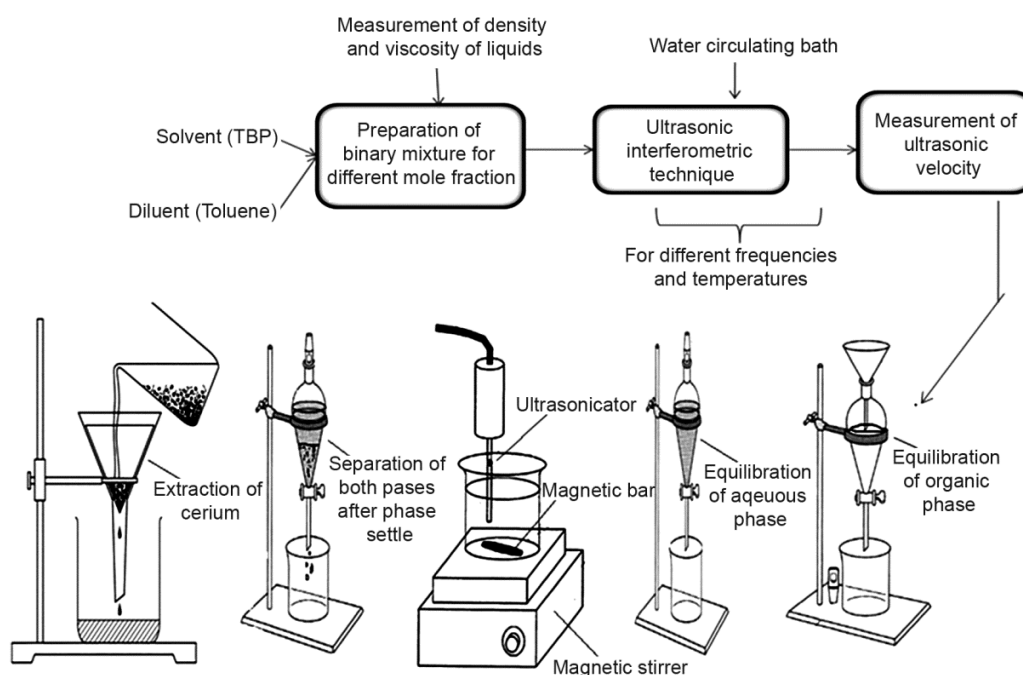


Fig. 1 — Schematic diagram for total extraction procedure of Ce from  $\text{CeO}_2$

equilibrated by adding tributyl phosphate with toluene of different mole fraction and aqueous phase was created by adding cerium oxide in diluted nitric acid using 25 mL flask. The biphasic system was put into the magnetic stirrer at 200 rpm for 10 min for better mixing of the solution (Fig. 1) and ultrasonicator was used for phase settle. After the phase settle, the extract was filtered through a 0.45  $\mu\text{m}$  nylon membrane (Guangfu Chemical Reagents Co., Tianjin, China). To verify the measured distribution ratios mass balance analysis was used by comparing the theory of chemical equilibrium according to Nernst distribution law:

$$K_D = \frac{[X]_{\text{org}}}{[X]_{\text{aqu}}} \quad \dots (1)$$

where the brackets denote the concentration of  $X$  in each phase at constant temperature. By principle, the concentration extracted into organic and aqueous phase appears in the numerator and denominator of equation.

#### 4 Theory

The transmission of acoustic wave through a treated medium is due to the mechanical vibrations or oscillatory motions of particles inside the medium. An ultrasonic wave may be visualized as an infinite number of oscillating masses or particles connected by means of elastic springs. Each individual particle is influenced by the motion of its nearest neighbor and both inertial and elastic restoring forces act upon each particle (Fig. 2).

Thus the force acting on the atoms of the medium as per hooke's law is given as:

$$F \propto -x \quad \dots (2)$$

$$F = -Kx$$

where,  $K$  is a constant which depends on the nature of the medium on which the wave propagates and the intermolecular interaction. As the liquid medium is elastic the wave equation given by:

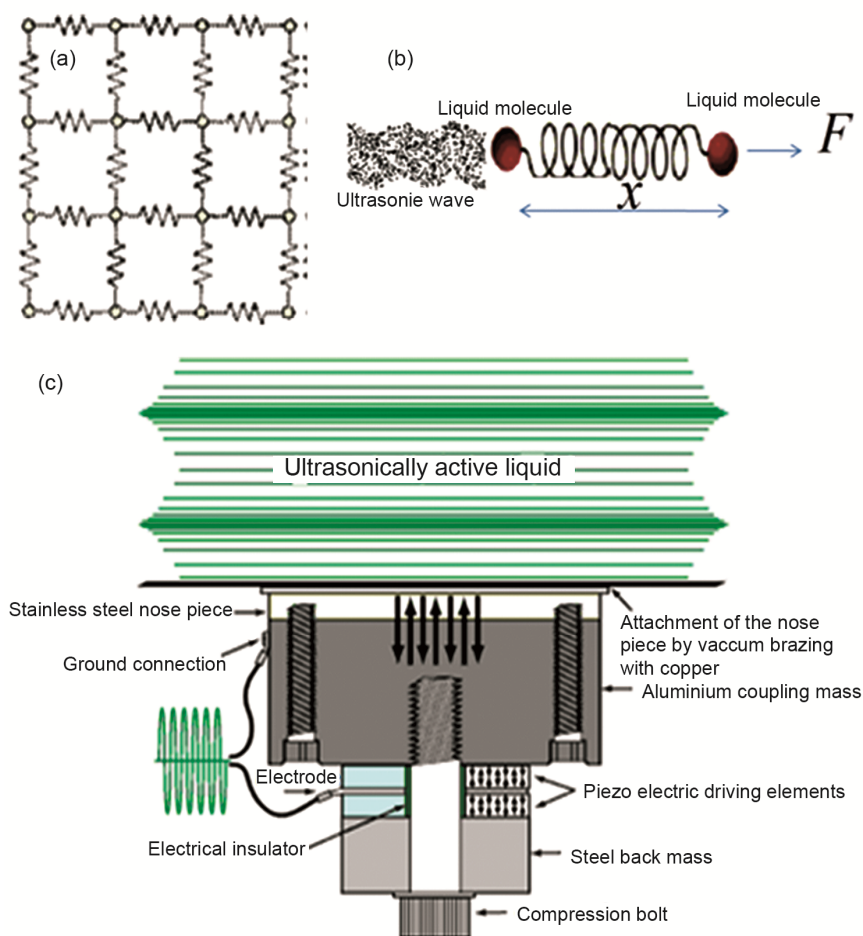


Fig. 2 — (a) Propagating medium, (b) force acting on propagating medium and (c) ultrasonically active medium

$$\frac{\partial^2 \Psi}{\partial x^2} = \frac{1}{C^2} \frac{\partial^2 \Psi}{\partial t^2} \quad \dots (3)$$

where  $C$  is the velocity of the wave or ultrasonic wave propagating through the medium. The experimental calculated values of ultrasonic velocity, density and viscosity are used to compute different acoustic parameters such as isentropic compressibility ( $\beta_s$ ), intermolecular free length ( $L_f$ ), acoustic impedance ( $Z$ ) and surface tension and their excess values which are well describing the physic-chemical properties of the medium. The acoustic parameters have been evaluated with the help of the following relationships<sup>7-10</sup>.

Isentropic compressibility:

$$\beta_s = \frac{1}{\rho C^2} \quad \dots (4)$$

Intermolecular free length:

$$L_f = k\beta^{1/2} \quad \dots (5)$$

Acoustic impedance:

$$Z = \rho C \quad \dots (6)$$

Surface tension:

$$\sigma = 6.4 \times 10^{-3} \rho C^{\frac{3}{2}} \quad \dots (7)$$

and their excess values are calculated as:

$$(Y^E) = Y_{\text{mix}} - (X_A Y_A + X_B Y_B) \quad \dots (8)$$

where  $X_A$  and  $X_B$  are the mole fractions,  $Y_A$ ,  $Y_B$  and  $Y_{\text{mix}}$  represent the isentropic compressibility, inter molecular free length, acoustic impedance, surface tension of toluene, TBP and their mixtures, respectively. The constant  $k$  is temperature dependent which is given<sup>11,12</sup> as :

$$[93.875 + (0.375T)] \times 10^{-8}$$

where  $T$  is denoted as absolute temperature.

## 5 Results and Discussion

In Table 1 experimentally calculated data<sup>3,13,14</sup> of density ( $\rho$ ) and viscosity ( $\eta$ ) of pure liquids are given. For various mole fraction of TBP,  $\rho$  and viscosity  $\eta$  values are presented in Table 2. The evaluated data of ultrasonic velocity ( $C$ ) and computed values of deviated acoustic impedance ( $Z^E$ ), deviated values of isentropic compressibility ( $\Delta\beta_s$ ), deviated intermolecular free length ( $L_f^E$ ) and excess surface tension ( $\sigma^E$ ) for a constant frequency 1 MHz and at different temperature 303.15 K, 308.15 K and 313.15 K are reported in Table 3 and 4, respectively. The percent of extraction for each mole fraction of TBP have been given in Table 5 which is calculated from Nernst distribution coefficient.

Table 1 — Experimental and literature values of density ( $\rho$ ) and viscosity ( $\eta$ ) for pure liquids at different temperatures

Component	$\rho$ (Kgm <sup>-3</sup> )			$\eta$ (Nsm <sup>-2</sup> )				
	Experimental		Literature	Experimental		Literature		
	303.15 K	308.15 K	313.15 K	303.15 K	308.15 K	313.15 K		
Tributyl phosphate	950.578	952.834	948.487	974.800 <sup>a</sup>	0.00178	0.00177	0.00175	0.0010092 <sup>a</sup>
Toluene	854.456	853.789	852.326	834.800 <sup>b</sup>	0.00159	0.00158	0.00156	0.0010086 <sup>c</sup>

<sup>a</sup>Reference<sup>3</sup> at 303.15 K, <sup>b</sup>Reference<sup>13</sup> at 308 K and <sup>c</sup>Reference<sup>14</sup> at 288.15 K

Table 2 — Experimentally evaluated values of density ( $\rho$ ) and viscosity ( $\eta$ ) at different temperatures for various concentration range of TBP

Mole fraction of TBP	$\rho$ (Kgm <sup>-3</sup> )			$\eta$ (Nsm <sup>-2</sup> )		
	303.15 K	308.15 K	313.15 K	303.15 K	308.15 K	313.15 K
0.0000	854.456	853.789	852.326	0.00159	0.00158	0.00156
0.0712	869.182	867.345	865.029	0.00157	0.00156	0.00154
0.1692	885.827	882.917	880.826	0.00155	0.00153	0.00152
0.2570	898.025	897.062	894.564	0.00154	0.00151	0.00149
0.4356	916.129	913.876	910.275	0.00157	0.00154	0.00153
0.5052	922.023	920.172	918.002	0.00168	0.00162	0.00159
0.5820	927.896	925.603	923.118	0.00171	0.00168	0.00164
0.7059	935.953	932.557	930.222	0.00173	0.00171	0.00168
0.7781	940.066	938.971	936.025	0.00175	0.00173	0.00171
0.8712	945.001	943.814	941.037	0.00176	0.00175	0.00173
1.0000	950.578	952.834	948.487	0.00178	0.00177	0.00175

Table 3 — Ultrasonic velocity ( $U$ ), deviated acoustic impedance ( $Z^E$ ), deviated isentropic compressibility ( $\Delta\beta_s$ ) of pure tributyl phosphate (TBP), toluene and binary mixture of TBP and toluene at different mole fraction with different temperature and constant frequency 1 MHz

Mole fraction of TBP	$U$ (m/s)			$Z^E$ ( $\text{kgm}^{-2}\text{s}$ ) $\times 10^4$			$\Delta\beta_s$ ( $\text{N}^{-1}\text{m}^{-2}$ ) $\times 10^{-7}$		
	303.15 K	308.15 K	313.15 K	303.15 K	308.15 K	313.15 K	303.15 K	308.15 K	313.15 K
X1									
0.0000	1277	1276	1275	0	0	0	0	0	0
0.0712	1273	1272	1271	8822.209136	8814.327022	11513.74148	-24.0977223	-24.0643520	-28.7016980
0.1692	1270	1269	1268	18398.69858	18383.59142	21064.14548	-40.2538249	-40.1950756	-44.6917156
0.2570	1266	1265	1264	22275.00696	22256.14131	24922.51257	-51.6533634	-51.5793962	-55.9574868
0.4356	1263	1262	1261	26126.04457	26106.24231	28750.47214	-45.1060738	-45.0362228	-49.2082175
0.5052	1260	1259	1258	24444.13866	24425.13249	27061.89494	-44.9226509	-44.8545345	-48.9463415
0.5820	1257	1256	1255	22042.54496	22025.04796	24654.20127	-42.4159704	-42.3526318	-46.3588178
0.7059	1254	1253	1252	18040.51155	18026.86707	20645.1625	-30.3684272	-30.3214008	-34.1980569
0.7781	1252	1251	1250	14720.53797	14709.7205	17322.27077	-22.7984274	-22.7622536	-26.5647744
0.8712	1247	1246	1245	7666.794136	7659.990622	10265.50128	-18.6133625	-18.5872947	-22.2903309
1.0000	1244	1243	1242	0	0	0	0	0	0

Table 4 — Deviated intermolecular free length ( $L_T^E$ ) and deviated surface tension ( $\sigma^E$ ) of pure tributyl phosphate (TBP), toluene and binary mixture of TBP and toluene at different mole fraction with different temperature and constant frequency 1 MHz

Mole fraction of TBP	$L_T^E$ (m) $\times 10^{-7}$			$\sigma^E$ ( $\text{Nm}^{-1}$ )		
	303.15 K	308.15 K	313.15 K	303.15 K	308.15 K	313.15 K
X1						
0.0000	0	0	0	0	0	0
0.0712	-5.5956	-5.5922	-6.7041	1917.2819	1868.9384	2484.5379
0.1692	-9.3571	-9.3506	-0.1043	4205.7083	4091.7773	4701.4340
0.2570	-0.1207	-0.1207	-0.0314	5041.4163	4869.8224	5475.2530
0.4356	-0.1047	-0.1046	-0.1149	6332.9984	6045.7990	6644.3220
0.5052	-0.1047	-0.1046	-0.1148	5918.0402	5586.4178	6182.8770
0.5820	-9.9252	-9.9180	-0.1091	5365.6501	4985.1909	5579.5545
0.7059	-7.0718	-7.0662	-0.8043	4634.7341	4175.7464	4766.9878
0.7781	-5.2872	-5.2828	-6.2457	3956.2692	3451.7538	4041.4089
0.8712	-4.4291	-4.4264	-5.3715	2193.7445	1630.9619	2219.0110
1.0000	0	0	0	0	0	0

Density is a parameter which is defined as the measurement of solvent-solvent and solute-solvent interactions. For different temperature variation, density of the liquid mixture has been measured for the biphasic system of TBP with toluene. The value of density is maximum for the low concentration region of non polar liquids like toluene and minimum for high concentration region of toluene (Fig. 3). This type of phenomena occurs due to the shrinkage in the volume of the solvent mixture. When temperature increases density of the liquid remain decreases. This phenomenon of variation of density can be explained as structure breaker of the solvent when density decreases to a particular concentration and structure

maker of the liquid when density increases. Density of the mixture found to be increasing constantly with the increase in concentration of the polar solvent like TBP. It indicates occurrence of strong intermolecular interaction within the solvent mixture<sup>15,16</sup>. Gradual addition TBP with toluene decreases the volume of the solvent mixture which can be pointed out by the initial quick increase in density value as shown in the graph.

Viscosity is the ease with which the solution flows. From Fig. 4 it is observed that the value of viscosity decreases with the increase in the mole percent of TBP up to mole fraction 0.4 and then after this concentration range increases gradually with increase

Table 5 — % extraction of cerium from cerium oxide with various concentrations of extractant diluent pair (EDP) at different temperatures

Mole fraction of EDP	% extraction of Cerium at different temperatures		
	303.15 K	308.15 K	313.15 K
0.0000	15	13	11
0.0712	33	30	28
0.1692	69	62	60
0.2570	75	70	67
0.4356	88	75	60
0.5052	87	73	59
0.5820	84	71	57
0.7059	81	71	60
0.7781	79	70	61
0.8712	80	72	62
1.0000	82	73	61

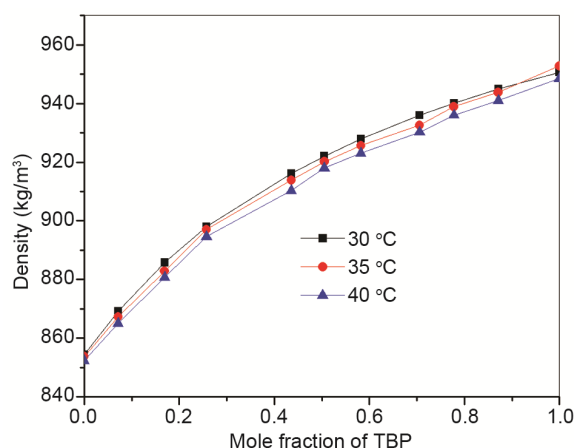


Fig. 3 — Variation of density with different mole fraction of TBP at different temperature

in mole fraction of TBP. Some agglomeration of complex formation in the primary stage is the main reason for this sort of activities. But if we observe its trends for different temperature, viscosity value decreases with rising temperature. TBP is highly viscous compared to toluene, so addition TBP with toluene influences the viscosity of the solvent mixture. The weak hydrogen bonding between the P=O oxygen in TBP and the methyl group present in toluene increase the resistance to flow and consequently viscosity increases<sup>17</sup>. For all frequency and temperature, the ultrasonic velocity of the binary mixture decreases linearly when concentration of TBP increases. This phenomenon is directed by the effect of density. The major reason of such discrepancy is due to difference in density value of toluene and TBP. There may be some dipole-dipole interaction present between the liquid<sup>18,19</sup>.

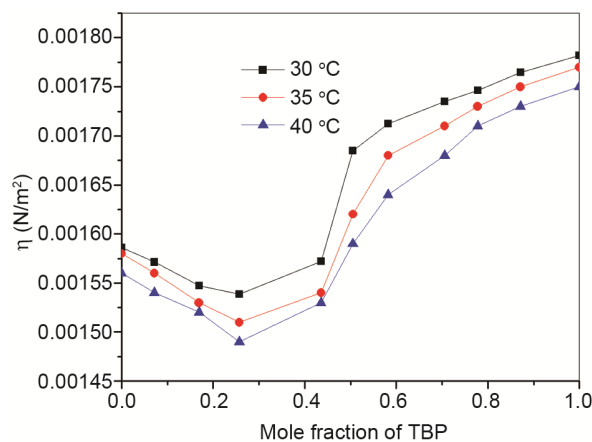


Fig. 4 — Variation of viscosity with different mole fraction of TBP at different temperature

Again, with increase of frequency and also for temperature the ultrasonic velocity decreases. This trend is recognized due to high oscillation of the solvent molecules caused by the dispersion of energy<sup>20-22</sup>. Due to the presence of P=O functional group in TBP molecule it is polar and therefore it can be able to interact with non polar molecules like toluene. Hence there may be polar-induced dipolar interactions occurring in the mixture. In pure TBP molecule there is dipole-dipole as well as the dispersive interaction present. When diluent like toluene is added to TBP, it initially displaces the structure of the TBP molecule. Velocity decreases with increasing concentration due to strong molecular interactions (Fig. 5(a, b)).

The deviation in the physical property from its ideal behavior is a measure of the degree of interaction between components molecules of the solvent mixture<sup>9</sup>. The variation in excess viscosity gives a quantitative estimation of the strength of intermolecular interactions and these values can be explained by considering two factors. One is the difference in size, and shape of the component molecules and the loss of dipolar association in pure compound that may lead to a decrease in viscosity. The other factor is the specific interactions between unlike components such as hydrogen bond formation and charge transfer complexes which may cause increase in viscosity in mixtures over that found in pure components. For the studied sample,  $\Delta\beta_s$  and  $L_r^E$  are negatively deviated (Fig. 6(a, b) and Fig. 7(a, b)) which pointed out that the solvent mixture is less compressible than the pure liquids forming the complex and molecules are more tightly

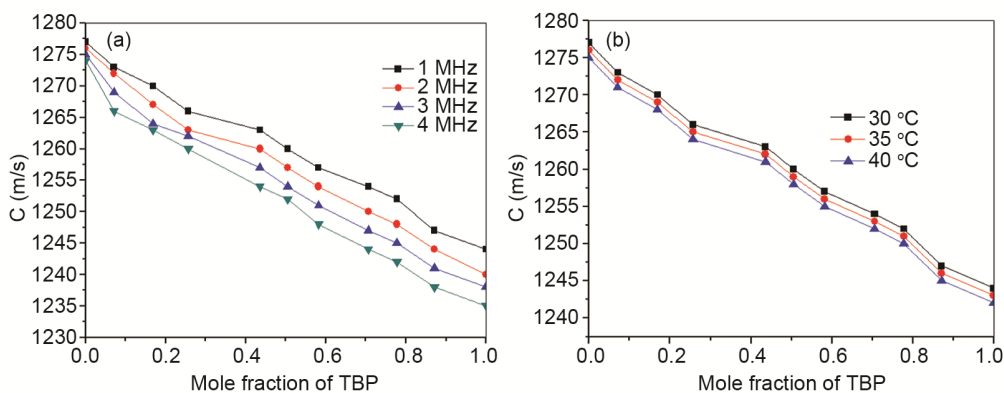


Fig. 5 — Variation of ultrasonic velocity with TBP at (a) different frequency and (b) temperature

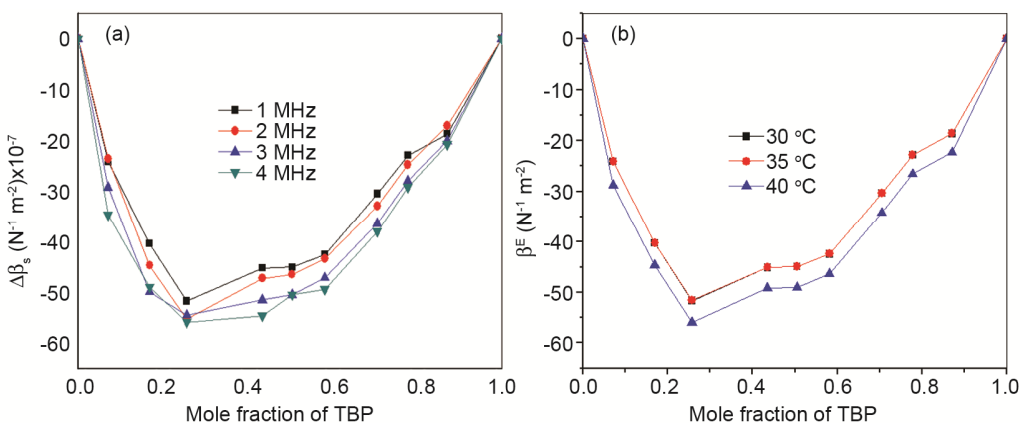


Fig. 6 — Variation of deviated isentropic compressibility with TBP at (a) different frequency and (b) temperature

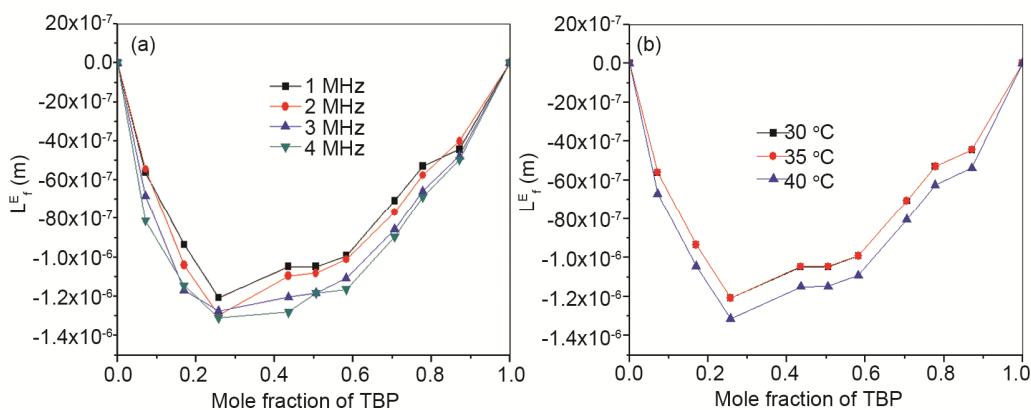


Fig. 7 — Variation of excess intermolecular free length with TBP at (a) different frequency and (b) temperature

bound than in pure liquids. This indicates the presence of relatively stronger molecular interaction, possible through hydrogen bonding<sup>25,26</sup> between the component molecules. Mixing of TBP with toluene will induce the breaking up of the associated clusters of TBP releasing several dipoles which in turn can

induce a dipole moment in toluene molecules, resulting dipole-induced dipole interactions. Thus, toluene acts as a structure breaker of TBP. With the increase in frequency the interaction becomes weak and the breaking of associated structure of TBP dominates, resulting in increase<sup>21</sup> of  $\Delta\beta_s$ . The result

shows same trend for increasing temperature also. Always low temperature (preferably room temperature) and low frequency are appropriate for extraction which is proved from analysis of the data. The negative values of  $L_T^E$  are attributed to close packaging of the component molecule decreases up to 0.3 mole fraction of TBP and then increases negatively attributing to loose packing of the component molecules which in fact supports the heteromolecular association between the TBP and toluene.

This results the decrease in free length between the component molecules which indicates that the system is more resistant to the sound propagation. The positive excess acoustic impedance ( $Z^E$ ) value shows that over the concentration range of 0.4-0.5 mole percent for TBP significant interaction between the molecules of solvent is present as shown in Fig. 8(a, b). This is recognized due to interaction between component molecules and formation of closer molecular aggregates which leads to decrease in the intermolecular free

space. This activity occurs due to phosphoryl group of tributyl phosphate and  $\text{CH}_3$  group of toluene. The deviated surface tension is also positively deviated during the total experimental process for all frequencies and temperatures. The positive deviated value specifies that increase in molar volume indicating strong interaction between the solvent molecules. The maximum deviation is seen at 0.4 mole fraction of TBP (Fig. 9(a, b)). The positive excess surface tension  $\sigma^E$  of mixture is due to the adsorption of TBP at the interface of the toluene and TBP. The lone pair of electrons on oxygen atom of the phosphate bond is responsible for this adsorption<sup>27</sup>. Low frequency and low temperature give significant result.

By the thorough analysis of physico-chemical parameters obtained from ultrasonic study of solvent mixtures makes it appropriate for their compatibility up to a particular concentration (0.4 mol% of TBP) for extraction of rare earth materials. TBP is a polar liquid having dipole moment  $\mu = 2.92$  D which has

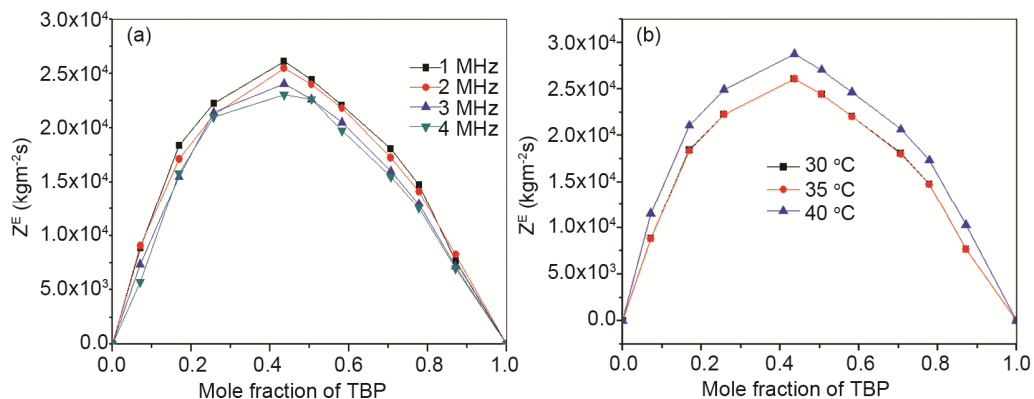


Fig. 8 — Variation of excess acoustic impedance with TBP at (a) different frequency and (b) temperature

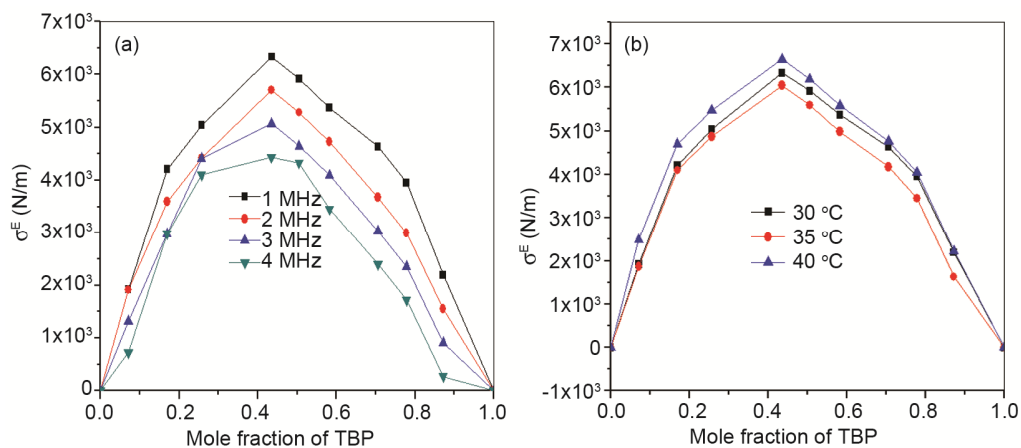


Fig. 9 — Variation of excess surface tension for different mole fraction of TBP at (a) different frequency and (b) temperature



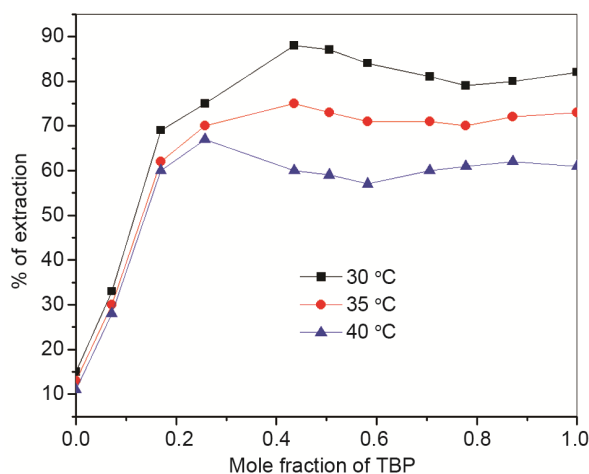


Fig. 10 — % of extraction for different mole fraction of TBP at different temperature

dielectric constant  $\epsilon = 8.29$ . Generally TBP is known to be a mildly associated polar liquid having linear<sup>28</sup> correlation factor ( $g > 1$ ). It can remain also in head tail arrangement with  $\alpha$ -multimerization. After the addition of non polar liquid such as toluene  $\alpha$ -multimers breaks which assists the solvent extraction phenomena. To carry out the extraction process each concentration of TBP/toluene was treated with  $\text{CeO}_2$  which is previously added with  $\text{HNO}_3$  and using Nernst distribution law the distribution coefficient was evaluated. The extraction of cerium increased gradually with the increase of TBP concentration up to 0.4 mole fraction and then plateaued as given in Fig. 10. But when temperature increases extraction efficiency remains decrease. For low temperature such as 303.15 K, it is giving excellent result for extraction. However diluent are unable to extract metal ion from the aqueous phase, but they greatly affect the extraction behavior of extractants<sup>29</sup>.

## 6 Conclusions

By the application of ultrasonic technique in the liquid mixture of TBP and toluene, different types of molecular interaction present inside solvent mixture are analyzed. From the experimentally evaluated acoustic data of ultrasonic velocity, density, viscosity and acoustic parameters with their deviated values of TBP-toluene with various concentrations range indicate the presence of significant molecular interactions within the studied liquid solution. Acoustical parameters are also influenced by different factors like frequency, temperature and environmental condition, which is confirmed from the investigation of graph, plotted using the acoustical parameters. The

alteration of deviated physico-chemical parameter from particular mole fraction suggests that TBP is compatible with toluene for extraction cerium from cerium oxide. An optimized value for the extraction is found out by examining the nature of interaction come out after the propagation of ultrasonic wave. From the overall investigation it is confirmed that TBP with toluene may be used as efficient diluents and the extraction efficiency of cerium is found to be 80-85% for low temperature and low frequency ultrasonic wave.

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