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Ultrasound-assisted of alkali chloride separation using bulk ionic liquid membrane

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ABSTRACT

An ultrasonic-assisted separation of alkali chloride (LiCl, NaCl, and KCl) salts have been carried out using of an hydrophobic ionic liquid membrane (ILM). The ionic liquid 1-butyl-3-methylimidazolium hexafluorophosphate and tributyl phosphate mixture have been used as ILM. An ultrasonic probe with different frequencies (25, 100, and 250) kHz have been applied as source of ultrasound generator with different times of sonication (2, 5, and 10) min in three phases system containing feed, ILM, and receiver in osmotic U-shaped tube. Also, 250, 500, and 1000 ppm of the feed (alkali chloride) concentration have been used to separate. The frequency of 250 kHz with higher sonication time provides optimum condition for separation of LiCl with lower feed concentration. The thermodynamic properties such as density and speed of sound and the related thermodynamic properties have been calculated to optimize ILM composition ($x_{IL} = 0.45$) for ultrasound-separation.

1. Introduction

The separation of alkali metals is one of the most important industry issues. There are numerous methods for the separation and extraction of these metals. [1–3] Recently, lithium has been separated from spent battery material using ultrasonic leaching with an efficiency >99%. [4] However, using a membrane is one of the most successful and environmentally friendly methods. The membrane technology was applied in various applications such as filters, batteries, and artificial cell membranes. [5–8]

Recently, ionic liquids (ILs) as green and novel materials in the membrane's separation technologies have been raised. These compounds have the potentials to apply in different parts of the membrane separation technologies such as the striping phase, membrane phase, and receiving phase, etc. [9,10] Most recent investigations have demonstrated that hydrophobic ILs in the presence of the hydrophobic organic compounds could be used as a membrane for the separation of a water-soluble organic compound such as carboxylic acids. [11,12]

The use of ultrasound wave for separation and extraction of various materials have been developed as a green and environmentally friendly technique in the last years. [13,14] Physical treatment instead of the toxic chemicals on the industrial scale is the privilege of the ultrasound in the processes. [15–17] More green process for separation could be achieved using ultrasonic in the membrane technology.

The theory of separation with ultrasonic is based on particle displacement under sound pressure with a semipermeable liquid membrane phase. Numerous water-immiscible components could be used as a liquid membrane for the separation of water-soluble contents. In this work, the hydrophobic ionic liquid 1-butyl-3-methylimidazolium hexafluorophosphate as the base of the ionic liquid membrane has been selected in the presence of tributyl phosphate as a carrier to adjust the ILM properties in separation. To achieve an appropriate separation level of LiCl, NaCl, and KCl from an aqueous solution of these salts with ultrasonic pressure, different compositions of the [BMIM][PF_6] + TBP have been investigated. Also, frequency, sonication time, and the thermodynamic properties of the ILM have been studied to analyze the parameters that affect the separation.

2. Experimental

2.1. Chemicals

All of the compounds are used in this work have been purchased from Merck. The used materials have listed in Table 1 with briefed information. The water used in this work is doubly distilled water with specific conductance less than 1 μS cm^{-1}.

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Table 1
A summary of the chemicals and their characteristics that were used in this work.

Chemical name	CAS number	Abbreviation	Supplier	Purity w/w	Purification method	Water content (ppm)	Final mass fraction purity	Analysis method
1-Methylimidazole	616–47-7		Merck	>0.99	Distillation			
1-Chlorobutane	109–69-3		Merck	>0.99	None			
Ethyl acetate	141–78-6		Merck	>0.995	None			
Tributyl phosphate	126–73-8	TBP	Merck	>0.99	None			
Dichloromethane	75–09-2		Merck	>0.99	Dean-Stark			
Lithium chloride	7447–41-8		Merck	>0.99	None			
Sodium chloride	7647–14-5		Merck	>0.995	None			
Potassium chloride	7447–40-7		Merck	>0.995	None			
Potassium hexafluorophosphate	17084–13-8		Merck	>0.99	None			
1-Butyl-3-methylimidazolium hexafluorophosphate	174501–64-5	[BMIM][PF_6]	Synthesized		Extraction / filtration / rotary evaporator / vacuum	50	>0.97	H'NMR / KF

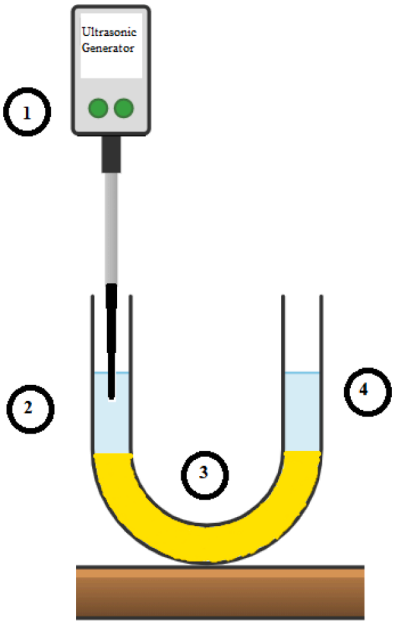

Fig 1. (1) Ultrasonic probe, (2) feed phase, (3) membrane phase, and (4) receiving phase.

2.2. General procedure for the synthesis of [BMIM][PF_6]

The 1-butyl-3-methylimidazolium chloride [BMIM]Cl was prepared from 1-methylimidazole and 1-chlorobutane at 70 °C. Then, 1-butyl-3-methylimidazolium hexafluorophosphate has been synthesized with an anion exchange reaction between initial {BMIM]Cl and KPF_6 in the aqueous media at 25 °C. The solvents used to purify the ILs were ethyl acetate and dichloromethane for step 1 and step2, respectively. The synthesized [BMIM][PF6] has been characterized by ^{1}H NMR to ensure the absence of any impurity.

2.3. Ionic liquid membrane preparation

The two-component ionic liquid membrane (ILM) was prepared in different concentrations of the IL and tributyl phosphate (TBP) by weighing with an analytical balance (Shimadzu, 321–34553, Shimadzu Co., Japan) with a resolution 10^{-8} kg. The mixture was stirred to form a homogenous liquid. Also, the conservation of mass in the membrane is necessary for the methods which are used in this study for the determination of the separation amount.

2.4. Density and speed of sound measurement

An oscillating U-shaped instrument (Anton Paar, DSA 5000, Austria) has been used to determine the density and speed of sound of the prepared ILM. This instrument has calibrated by air and deionized water. The sound waves are used to measure the density and speed of sound have a frequency of 3 MHz. The resolution of the instrument is 0.001 kg m^{-3} for density and 0.01 m s^{-1} for speed of sound. Also, the uncertainty of the instrument has been investigated and reported in the literature. [18]

2.5. Separation of alkali chloride salts with ultrasound and ILM

A U-shaped tube (osmosis tube) was used as a process vessel as tabulated in Fig. 1. The ultrasonic probe was used with different frequencies as a source of sound pressure to separate the alkali chloride salts from an aqueous media with a bulk ionic liquid membrane with different compositions and at a different time of sonication process.

The feed phase and receiving phase have been charged at the same time from the different sides of the osmosis tube with a controllable and low rate of flow. It is necessary to prevent turbulence and no major displacement of the ILM which may cause mixing of the phases and concentration gradient. While a relatively large amount of the ILM could prevent turbulence and concentration gradient, the small amount is appropriate for effective separation.

An ultrasonic probe (Dr. Heilsher, GmbH, UP-400, ultraschall-prozessor) with the adjustable time interval of sonication, amplitude, and frequency was used to sonicate and separate LiCl, NaCl, and KCl (1000 ppm, 500 ppm, and 250 ppm) with ILM. The sonication process was carried out for 2, 5, and 10 min. Also, applied frequencies are 25, 100, and 250 kHz. It should be noted that in the case of applying 550 kHz, TBP was dispersed in the feed phase, and the membrane composition was destructed. The ultrasound has been applied with 1-second interval pulses with a 20% amplitude. Also, to prevent heating of the system during the sonication an ice bath with a temperature 293.15 K is used effectively.

The receiving phase analyzes for separated components (Li, Na, and K) has been carried out with a flame photometer (Jenway, PFP7). It should be noted that the calibration of the instrument was done with standards determined by the manufacturer. Briefly, 1000 ppm of aqueous KCl, NaCl, and LiCl has been prepared and diluted to less than 10 ppm. Also, the prepared stocks have been used as feed phase.

2.6. ILM recycling process

To recycle the prepared ILM after the separation process, double distilled water is used to wash the mixture of ILM. This process had carried out in two steps. In the first step, a mechanical and magnetic stirrer was used to stir the mixture for 5 min and aqueous phases have decanted. In the second step, an ultrasonic bath and mechanical stirrer

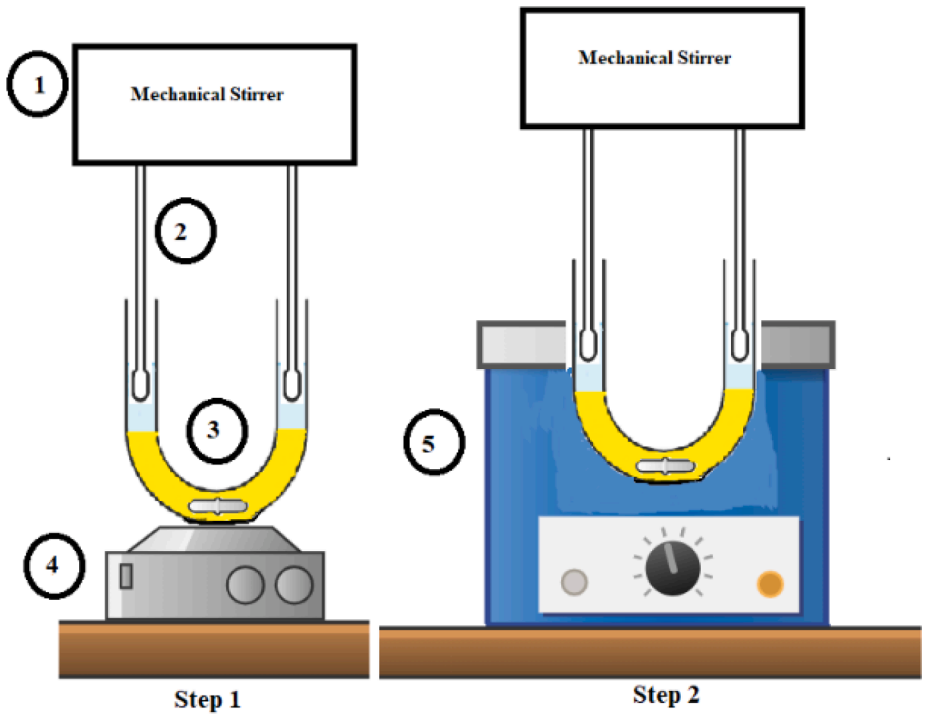

Fig 2. Schematic of recycling setup for ILM with deionized water; (1) mechanical stirrer with controlled speed, (2) glass stirrer rod, (3) magnetic bar, (4) magnetic stirrer, and (5) ultrasonic bath.

were used to mix and disperse the solution for 2 min and the aqueous phases have decanted. It should be noted that the solubility of water in the component of the ILM is negligible. The process is demonstrated schematically in Fig. 2. Also, recycled ILM has been weighted to ensure mass conservation. However, less than 2% of the weight fraction of ILM on average is lost in each process.

3. Results and discussion

3.1. Theoretical background of ultrasound

Sound defines as a sequence of waves of local pressure deviation from ambient pressure that propagates through compressible media. The total pressure in the presence of the sound could be defined as a sum of ambient pressure as static pressure, P_S, and a dynamic oscillation pressure, P_D, of sound P (P_S, P_D). This phenomenon may lead to the displacement of a particle between two immiscible mediums and consequently separation processes. The sound pressure is a complex variable that depended on time and position (t and x). The analytical expressions for the pressure could be evaluated through a physical–mathematical process in one dimension; [19]

$$P(x,t) = Aexp[j(\omega t - kx)] + Bexp[j(\omega t + kx)] \quad (1)$$

where, P, x, and t represents sound pressure, position, and time respectively. Also, ω is the angular frequency, k is the wavenumber, and j represents the imaginary number (j^2 = -1). The sound pressure time dependency when the position is fixed makes a harmonic oscillation which is the real part of the pressure that is dependent on time. Also, for the fixed time the mean square sound pressure which is a resultant of multiplying pressure on conjugated complex numbers could be evaluated as the magnitude of sound pressure that depended on position x and is a real and positive value. So, pressure varies with *cos (2kx)* and *sin (2kx)* as a result of maximum and minimum interference. This result could be generalized for 3 dimensions. It means the stationary pattern will be obtained for pressure in space, but the phase of pressure would not be the same in all positions. The applied sound pressure would cause a particle displacement. The general solutions for particle displacement in positive and negative-going waves take the same form. So, it convinces that particle displacement could be determined by positive-going wave with this equation; [19]

$$\xi^{+}(x,t) = h(ut - x) \quad (2)$$

u is the speed of sound and h represents the direction vector of the sound propagation. The time differentiation of the equation gives the acoustic particle velocity; [20,21]

$$v^{+}(x,t) = h'u \quad (3)$$

Also, differentiation with respect to the x gives expression for pressure:

$$p^{+}(x,t) = \gamma P_0\left(\frac{\partial \xi}{\partial x}\right) = h'\rho u^2 \quad (4)$$

Where γP_0 is equivalent to bulk modulus which could be evaluated by isentropic compressibility. It should be considered that there is no phase shift between acoustic particle velocity and sound pressure. Also, the magnitude of these variables determines acoustic impedance; [21]

$$\frac{|p^{+}(x,t)|}{|v^{+}(x,t)|} = Z_0 = \rho_0 u \quad (5)$$

Here, ρ_0 is the density of media and Z_0 is acoustic impedance. According to the equation a particle would be displaced under a sound pressure p by velocity v. As clear from the equation, displacement of a particle is related to characteristics of media.

3.2. Separation of alkali chloride salts with ultrasound

It is necessary to canalize the sound waves to apply ultrasonic pressure as a driving force. In this respect, a U-shape osmosis tube with a 10 mm internal diameter (Fig. 1) was used which is appropriate to the ultrasonic probe and narrow enough to avoid most of the refraction, reflection, and diffraction of the propagated sound waves. To find out the ability of the separation system, different frequencies of ultrasound

Table 2
The analysis of components in feed phase, membrane phase, and receiving phase after ultrasonic treatment for Li, Na, and K in a different frequency, f, different membrane composition, x_{IL}, variate times, t, and different concentration of feed, c.

Variable	Li			Na			K		
	F phase	M phase	R phase	F phase	M phase	R phase	F phase	M phase	R phase
	ppm								
	$x_{IL} = 0.45$ / $t = 2$ min / $c = 1000$ ppm								
$f = 025$ kHz	928	43	29	946	33	21	962	26	12
$f = 100$ kHz	912	35	53	932	33	35	953	26	21
$f = 250$ kHz	899	29	72	912	36	52	946	20	34
	$x_{IL} = 0.45$ / $f = 25$ kHz / $c = 1000$ ppm								
$t = 02$ min	928	43	29	946	33	21	962	26	12
$t = 05$ min	883	58	59	922	33	45	948	31	21
$t = 10$ min	852	50	98	885	29	86	933	31	36
	$x_{IL} = 0.45$ / $f = 25$ kHz / $t = 2$ min								
$c = 0250$ ppm	106	20	124	148	14	88	201	17	32
$c = 0500$ ppm	350	38	112	432	23	45	462	20	18
$c = 1000$ ppm	928	43	29	946	33	21	962	26	12
	$f = 25$ kHz / $t = 2$ min / $c = 1000$ ppm								
$x_{IL} = 0.25$	895	25	80	923	27	50	932	27	41
$x_{IL} = 0.45$	928	43	29	946	33	21	962	26	12
$x_{IL} = 0.75$	965	23	12	962	30	8	976	19	5

F phase: feed phase, M phase: membrane phase, and R phase: receiving phase. (M phase = c – R phase – F phase)

with multiple separation times and diverse concentration of the feed phase in various compositions of ILM were applied under atmospheric pressure at room temperature (298.15 K). Also, frequency has been chosen in a range (25 – 250) kHz to avoid attenuation of the ultrasound waves crossing from the osmosis tube. [22,23] The results for the separation amount of the alkali chloride salts (LiCl, NaCl, and KCl) under various conditions are given in Table 2.

3.2.1. Effect of ultrasonic frequency in separation

To understand the effect of frequency on the separation of LiCl, NaCl, and KCl with ultrasonic pressure by ILM, a constant composition of ILM with $x_{IL} = 0.45$ was selected. Also, 1000 ppm of each salt aqueous solution has been used as a feed phase. The ultrasonic has been applied for 2 min with 25, 100, and 250 kHz frequencies. As clear from Table 2, increasing in frequency enhances the separation generally. Also, the results show that Li could be separated effectively rather than Na and K. It seems that the size of the particle has a reverse relation with displacement. The speed of sound has a larger value for the NaCl rather than KCl. [24] To compare obtained results with concentration gradient treatment, the same concentration of the feed solutions has been charged in the system and after 4 h the content analysis has been carried out and a negligible amount of alkali chloride was separated compared with ultrasonic treatment.

3.2.2. Effect of sonication time in separation

Time is an essential matter of efficiency for a separation process. In this investigation, the effect of time has been compared with a concentration gradient versus ultrasonic treatment for alkali chloride metals separation. In this respect, the concentration gradient has been studied beside the ultrasonic treatment. The results of the analysis for the concentration gradient of the alkali chloride salts by using the investigated ILM was less than 2 ppm after 5 h. The results of the ultrasonic treatment in different process times are reported in Table 2 which indicates a considerable increase in the separation amount. The raised sonication time increases the separation amount. The attenuation of sound in the media with lower density is the main reason for the reduced amount of separation at lower sonication times in the processes. [25] The ultrasound increases the separation rate considerably. A Comparison of separation time for lithium with a successful selective separation of lithium with ionic liquid membrane with similar system show that it is slower than this method. [26]

3.2.3. Effect of feed concentration

Investigation upon the concentration of the studied aqueous alkali chloride contains useful information about ultrasonic propagation and its effect on particle displacement. The applied method is more effective in dilute solutions. It could be interpreted with the higher particle density which could fill the minimum pressure location. [27] However, it is an obvious fact that the speed of sound is higher at dense media rather than low-density media and lithium would be streamed more than sodium or potassium. Usually, in the conventional membrane separation higher flux of aimed component would be achieved with higher feed concentration of that component while in this method the reverse trend observed. For example, the results shows that 50% of the lithium with feed concentration of 250 ppm have been separated in 2 min while literature shows that 25% of the lithium have been separated from the feed with concentration of 600 ppm.[26] Also, raising feed concentration in this method lead to decrease in separation with same sonication time.

3.2.4. Effect of composition of ILM

According to the reported data, the rate of separation has been decreased by an increment of the IL content in the ILM phase. The ionic liquid has a high viscosity which TBP has used to balance its effect. Indeed, the TBP was used as a carrier in the supported ionic liquid membrane. [28] Also, the sound could be absorbed by media with high viscosity. [29] The characteristics of the ILM phase are an important issue that could control the separation process. The thermodynamic properties of the ILM phase could be used to interpret the existing interactions between components. Also, it is useful to find out the bulk modulus behavior of the ILM.

3.2.5. Possible mechanism for ultrasonic separation

The ultrasound waves propagation directly depends on the media characteristics (three media were studied in the present work). In this study, cavitation during sonication can considerably affect the mass and heat transfer. Ultrasound waves help to creates cavitation bubbles which led to accelerated mass transfer of the solute to the boundary of membrane phase. After, it can occur the transportation of the solute to the receiver phase. Also, there is an increase of heat in the medium and cooling of the system during the sonication process is necessary. [30,31]

3.3. Thermodynamic framework

The density and speed of sound of the mixture containing [BMIM][PF_6] and tributyl phosphate (TBP) have been measured for the full range of composition under atmospheric pressure at T = (288.15 – 318.15) K with interval 10 K. These data are collected and compared

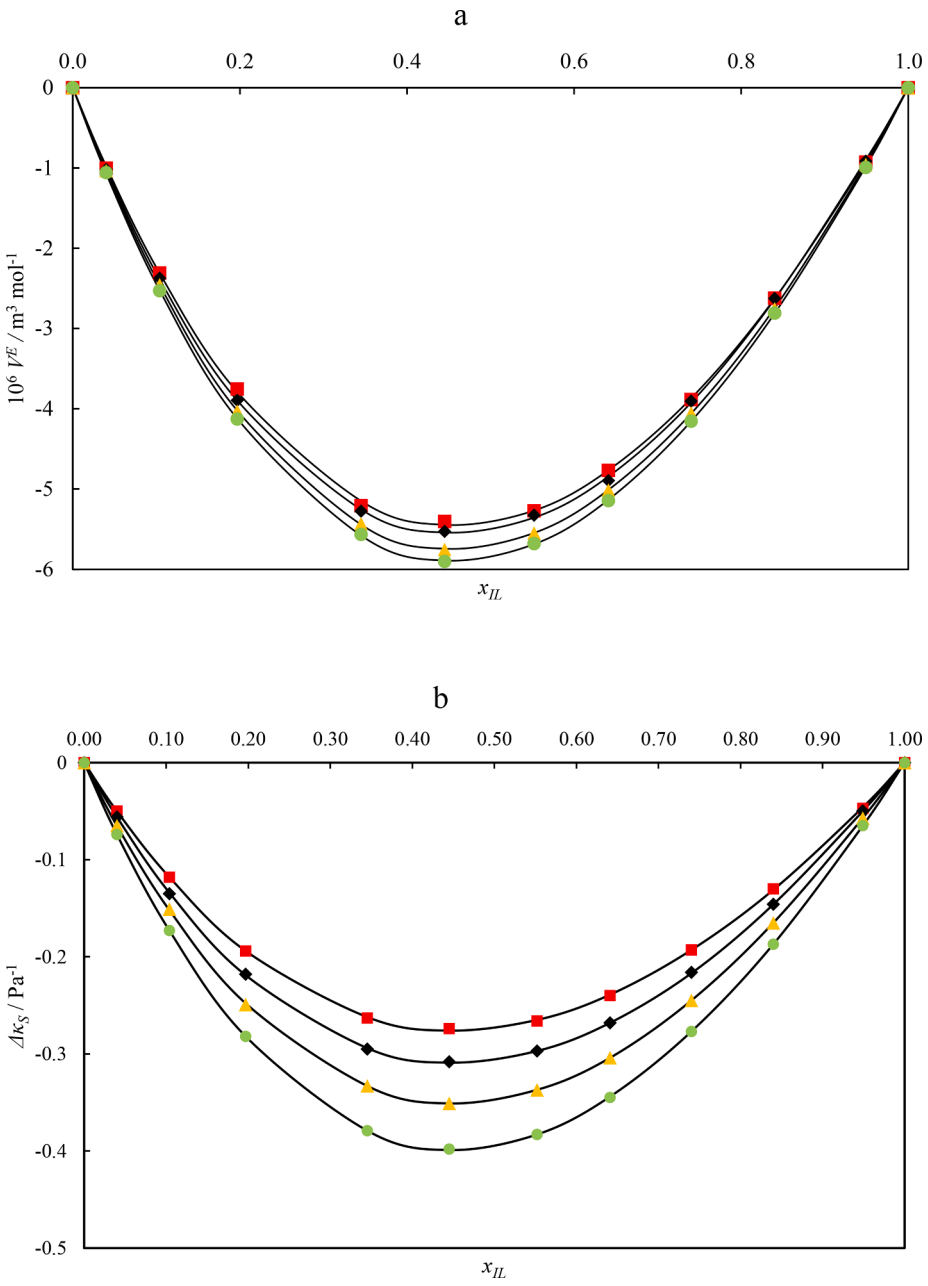

Fig 3. (a) The excess molar volume values and (b) Variation of isentropic compressibility deviations of the binary mixture of [BMIM][PF_6] + TBP versus the IL mol fraction at different temperatures: ■ 288.15 K, ◆ 298.15 K, ▲ 308.15 K, ● 318.15 and solid lines are corresponding Redlich-Kister values.

with literature [32] in supplementary materials. The excess molar volume, isentropic compressibility, deviation of isentropic compressibility, apparent molar volumes of the IL and TBP, and apparent molar isentropic compressibility are calculated by the following equations, respectively; [33,34]

$$V^E = \sum_{i=1}^{2} x_i M_i \left(\rho^{-1} - \rho_i^{-1}\right) \tag{6}$$

$$\kappa_s = \frac{1}{\rho u^2} \tag{7}$$

$$\Delta\kappa_s = \kappa_s - \sum_{i=1}^{2} x_i \kappa_{s_i} \tag{8}$$

$$V_\varphi = \frac{M}{\rho} - \left(\frac{\rho - \rho_0}{m\rho\rho_0}\right) \tag{9}$$

$$\kappa_\varphi = \frac{\kappa_S\rho_0 - \rho\kappa_{S_0}}{m\rho\rho_0} + \frac{\kappa_S M}{\rho} \tag{10}$$

where, x_i demonstrates mole fractions, and ρ_i, and ρ are densities of the pure components and the mixtures, respectively. M_i is molar mass of components and subscript $i = 1$ is for the TBP and $i = 2$ is for the [BMIM][PF_6]. The symbol u is used for speed of sound, κ_{s_i} is the isentropic compressibility for the pure components and κ_s is the mixture isentropic compressibility. Also, in the apparent molar properties, M (mol kg^{-1}) is the molar mass of the solute, m (mol kg^{-1}) is the molality of the solute. ρ_0 and ρ (kg m^3) are the densities of the solvent (pure IL or TBP) and the solution, respectively, and κ_{S_0} is the isentropic compressibility of pure solvent. A fifth-order Redlich-Kister equation is used to correlate V^E and

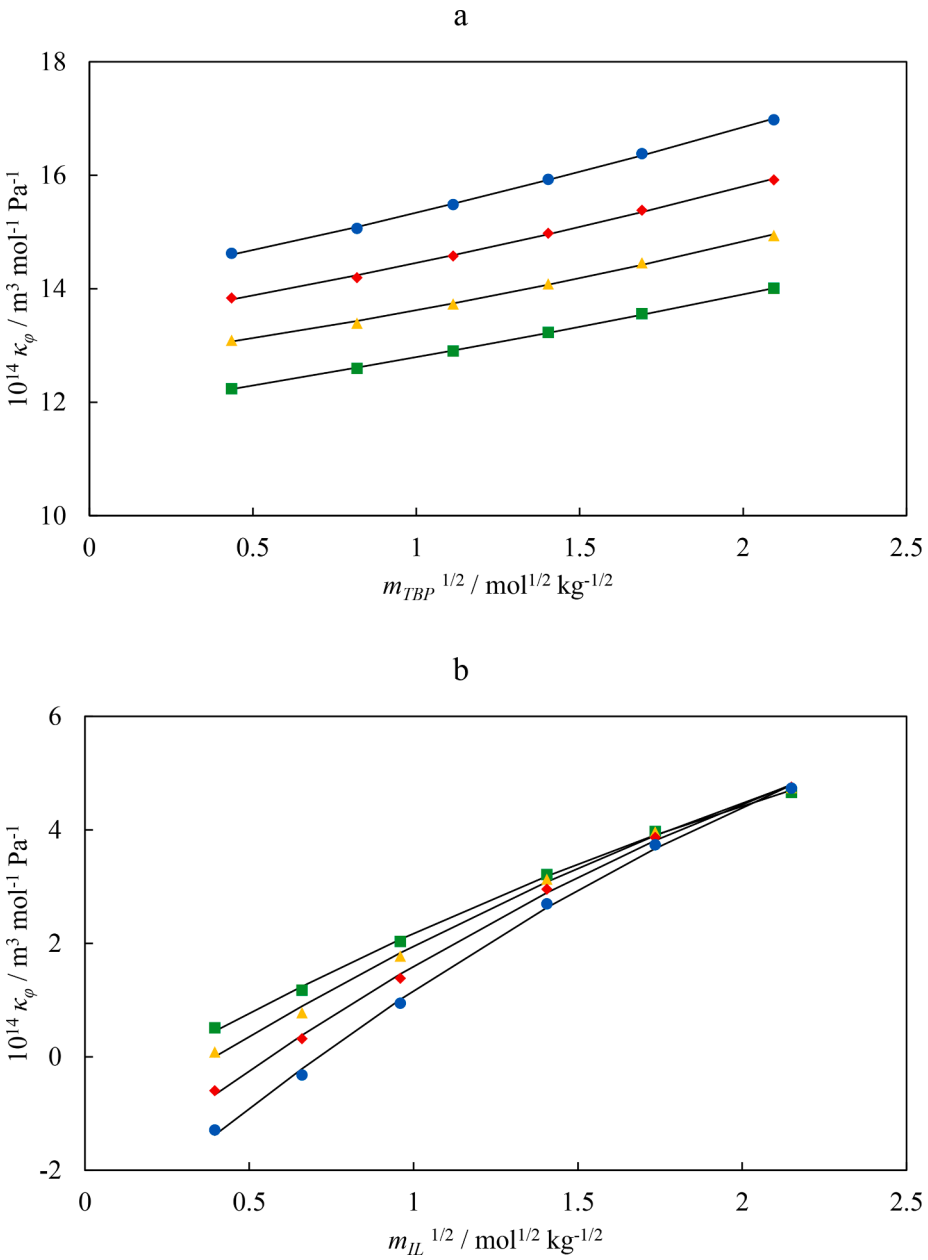

Fig 4. (a) The apparent molar isentropic compressibility values of TBP in [BMIM][PF_6] versus the TBP molality, and (b) the apparent molar isentropic compressibilities of [BMIM][PF_6] in TBP versus the IL molality at temperatures: ■ 288.15 K, ▲ 298.15 K, ♦ 308.15 K, ● 318.15 and solid lines are corresponding Redlich-Mayer values.

$\Delta\kappa_s$results. Also, Redlich-Mayer equation is used to correlate $V_\varphi$and $\kappa_\varphi$values. All of these results are given in supplementary materials Tables S1 and S2. Also, Figs. 3 and 4 containing important results that led to find best composition for ILM. The conceptual determination of these properties in find interactions and other physical issues are given in elsewhere. [33,34] The best composition is x_{IL} = 0.45 that makes proper membrane features for sonication process in separation.

4. Conclusion

The three-phase system containing [BMIM][PF_6] and TBP as ILM has been used to assess the ultrasonic effect on the separation of aqueous LiCl, NaCl, and KCl. The thermodynamic properties have been used to optimize the ILM composition under ultrasound. The best composition was obtained x_{IL} = 0.45. In the present work, the ultrasonic pressure as a driving force for the separation of the studied alkali chloride metals from aqueous media has been investigated. In this respect, the effect of the frequency, time, composition of ILM, and concentration of the feed phase has been studied. According to the results, ultrasonic could be efficient in separating alkali chloride metals from aqueous media with low concentration and frequency up to 250 kHz. Also, the small size metals (Li) separation was more efficient rather than the larger one (K). The high concentration of IL in the ILM phase may cause a decrement in separation efficiency but it is necessary to stabilize the ILM phase in the case of rising temperature during the use of ultrasonic.

CRediT authorship contribution statement

Hemayat Shekaari: Conceptualization, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Validation, Visualization, Writing - review & editing. **Behrang Golmohammadi:** Conceptualization, Data curation,

Investigation, Methodology, Software, Visualization, Writing - original draft.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Diagrams made in Chemix (https://chemix.org)

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ultsonch.2021.105549.

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