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Synthesis, characterization and electrochemistry of triethyl ammonium sulphate ionic liquid

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Abstract: Protic ionic liquids (PILs) being intrinsic proton conducting ionic species are considered as potential green electrolytes for study of electrocatalytic reactions and for fabrication of IL-based fuel cells (FCs) and batteries. We have prepared a sulfate anion based protic ionic liquid (PIL), triethylammonium sulfate (TEAS) through a reaction involving transfer of proton from H_2SO_4 to triethylamine (TEA). ^1H NMR and FT-IR spectroscopic techniques were employed for confirmation of the synthesis of TEAS and water content of the PIL was quantified using coulometric Karl–Fischer (KF) titration. ^1H NMR and FT-IR analysis confirm the synthesis of the PILs and KF-titration analysis shows that TEAS contains 1.43 w/w % water. Electrical conductivity of TEAS was determined at different temperatures showing that the PIL has excellent ionic conductivity that enhances with rise in temperature of the medium. The temperature dependence of the conductivity of the PIL follows the Arrhenius equation as the $\log\sigma$ versus $1/T$ plot is linear. The electrochemical windows (EWs) of the electrolyte were found using cyclic voltammetry at Pt and Au working electrodes and found to decrease with increase in temperature of the medium. The data revealed that the surfaces of the electrodes

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are covered with oxide layers due to oxidation of trace water (1.43 w/w %) present in the PIL. The oxide layers growth increase and their onset potential moves to less positive values as the temperature of the PILs is increased. The data was compared with the literature and would be helpful in understanding of the surface electrochemistry in this neoteric medium for being used as potential electrolyte in industry for various electrochemical applications.

Keywords: Arrhenius model; conductivity; electrolytes; electrocatalysis; electrochemical window; oxide layer; protic ionic liquids.

1 Introduction

Ionic Liquids (ILs) are salts which are liquid generally up to 100 °C [1, 2]. They are formed by organic unsymmetrical imidazolium, pyridinium, pyrrolidinium, trialkylammonium, quaternary alkyl ammonium, and tetraalkylphosponium cations and organic or inorganic nature anions such as methane sulphonate, trifluoromethane sulphonate, sulphate, bisulphate, nitrate, halide etc. Due to non-compact packing of these unsymmetrical cations and anions in the crystal lattice, resulting in the formation salts having melting points below 100 °C [3]. ILs may be protic ionic liquids (PILs) that contain conducting proton, formed by Bronsted acid-base reaction and aprotic ionic liquids (APILs) with no proton typically formed by irreversible hetero-atoms alkylation [4, 5]. The first RTIL reported was ethylammonium nitrate $[\text{EtNH}_3][\text{NO}_3]$ synthesized in 1914 by Walden by reacting ethyl amine with nitric acid that melts at 12 °C and is categorized as PIL. Later on alkyl imidazolium halogen aluminates ionic liquids were synthesized and were named as first generation ionic liquids and now classified as APILs. Since then a variety ILs of various types of cations and anions were synthesized and introduced in the literature [6, 7]. The synthesis of the PILs involve direct stoichiometric neutralization of proton donor species and proton acceptor species ($\text{HA} + :\text{B} \rightleftharpoons \text{BH}^+ + \text{A}^-$). The difference in aqueous pKa values, ΔpKa (pKa of base – pKa of acid) show how efficiently is the transfer of proton from acid to a base. Higher ΔpKa indicates good proton shifting from acid to base resulting in more thermally and electrochemically stable PILs. $\Delta\text{pKa} > 10$ between the proton donor acid and proton acceptor base can cause complete proton transfer and highly thermally stable IL can form [8–12].

ILs have intrinsic ions conductivities, high thermal and chemical stability (wide electrochemical window (EW)), less volatility and toxicity compared with organic and aqueous electrolyte [1, 12, 13]. They can be employed as neoteric solvents and electrolytes in various electrochemical processes including

fundamental investigations into mass and charge transport, studies of their electrical double layers, as electrochemical sensors and in batteries, supercapacitors and fuel cells [7,14–16]. ILs are proton conducting liquids that can be potential electrolytes for intermediate temperature FCs operating under non-humid conditions [7, 17, 18].

Here in we report the synthesis of a low cost sulphate anion based protic ionic liquid; triethylammonium sulphate (TEAS) and characterized through ^1H NMR, FT-IR and Karl–Fischer (KF) titration. Electrochemistry can be employed in many research fields and for different types of electrolytes used for different specific purposes [19–22]. The PIL was subjected also to electrochemical investigation through cyclic voltammetry to determined its EW at Au and Pt electrodes and examined the effect of temperature on the EW of TEAS. As far as we know, no electrochemical investigations have been carried out using triethylammonium sulphate as electrolytic medium and this is the main novelty of the work.

2 Experimental section

2.1 Reagents and apparatus

Analytical grade triethylamine, TEA (>99%) and H_2SO_4 (95–98%) were from Dae-Jung and Merck, respectively and used as received. Mettler Toledo Karl-Fischer (KF) titrator was used for the determination of water in TEAS and Bruker 300 MHz NMR instrument and PerkinElmer (UK). FT-IR spectrometer were used for confirmation of TEAS synthesis and its structure elucidation. Jenway (model 4510) conductivity metre (error bar $\pm 0.5\%$) was employed for measuring ionic conductivities of TEAS at various temperatures while an Autolab Potentiostat (Netherland) controlled from a computer was used for electrochemical measurements of the ionic liquid.

2.2 Procedure for TEAS preparation

The PIL was made from the precursor acid, H_2SO_4 and the base, $(\text{C}_2\text{H}_5)_3\text{N}$ (triethyl ammine) reaction taken in 1:2 by the procedures reported in literature [23]. A dry three-necked flask was charged with 34.9 ml (0.25 mol) of TEA fitted with a thermometer, a funnel with stopper and a condenser. The acid, 6.9 ml (0.25 mol) was added to TEA drop-wise from the funnel. Thermometer was used for monitoring temperature rise in the flask which was controlled through ice bath in which the sealed three-necked flask was immersed. The mixture was continuously stirred

through magnetic stirring. After all of H_2SO_4 was consumed, the reaction mixture was taken in a one neck round bottom glass flask and was subjected to further 24 h stirring on magnetic stirrer at about 60–70 °C for complete reaction (Scheme 1). TEAS was synthesized and was soft solid at room temperature but became viscous liquid at above 45 °C (melting point of TEAS).

2.3 Electrochemistry of TEAS

The electrochemistry of the TEAS mainly EW was probed at 2 mm diameter Pt and Au working electrodes in a conventional three electrode cell arrangement by cyclic voltammetry. The cell was charged with a 5 ml of TEAS and purged with N_2 for 20 min for deaerating the sample to remove electroactive dissolved O_2 . The working electrodes were polished on soft polishing pad using an aqueous suspension of 0.05 μm Al_2O_3 and immersed in the electrochemical cell along with Pt mesh (high surface area) counter and Ag wire reference electrodes. The cell was then placed on hot plate in an oil bath to study temperature effects.

3 Results and discussion

3.1 ^1H NMR of TEAS

Figure 1 depicts the ^1H NMR of TEAS taken in CDCl_3 as a reference solvent. The ^1H NMR spectrum of TEAS shows five peaks. A small peak in the spectrum at a chemical shift 7.280 ppm is observed that is attributed to the reference CDCl_3 . A large intense peak observed at chemical shift of 1.306 ppm (upfield chemical shift) labelled as 1 in the figure is attributed to the same environment nine protons of the CH_3 in $[\text{TEA}]^+$. The three protons of each of the three methy groups couples with the two protons of the methylene (CH_2) groups resulting in a triplet. A similar intense peak (less intense than peak 1) at an average chemical shift of 3.149 ppm (labelled as 2 in the spectra) corresponds to the six protons of the three methylene (CH_2) groups in the cation. This peak is quadruplet due to coupling with the three neighbouring protons of the CH_3 group. All these chemical shifts were observed at downfield compared to ^1H NMR spectra of pure triethylamine [24], which is an indicative of positive charge on nitrogen atom in TEAHS which pulls electron density from proton nuclei of carbon 1 and 2 and as a result experience greater

Figure 1: ^1H NMR spectrum of TEAS in CDCl_3 .

effective magnetic field. A peak at 6.415 ppm shift is observed which we attribute to the proton of the unreacted bisulfate $[\text{HSO}_4]^-$ ions. A small peak at a downfield chemical shift 10.675 ppm is seen which is not present in the ^1H NMR spectrum of triethyl amine (TEA) [24]. This corresponds to the proton directly attached to the nitrogen atom of the TEA and is due to transfer of proton from H_2SO_4 to the N of TEA and confirms the formation of the PIL.

3.2 FT-IR analysis of TEAS

The FT-IR spectra obtained for TEAS is shown in Figure 2 as % transmittance versus wave number (cm^{-1}). It is evident from the figure that at wave number higher than 2700 cm^{-1} we can see two set of absorption bands in the range around $2660\text{--}2880 \text{ cm}^{-1}$ and $3100\text{--}2900 \text{ cm}^{-1}$. The vibration due to CH stretching appears usually in $3100\text{--}2900 \text{ cm}^{-1}$ range [25] and stretching vibration bands in this wave number region can be seen in the FT-IR spectra of triethylamine [26]. Where the absorption peaks in $2660\text{--}2890 \text{ cm}^{-1}$ range that usually appear for NH stretching of ammonium cation [25] cannot be seen in the triethylamine FT-IR spectra [26]. Therefore, comparing our FT-IR spectra for triethylammonium sulphate ionic liquid with the FT-IR spectra of the triethylamine [26], the peaks within the range of $2660\text{--}2900 \text{ cm}^{-1}$ are attributed totally to NH stretching vibrations in the TEAS [25, 26]. The CH stretching vibrations in our TEAS spectra appear in the region of

Figure 2: FT-IR spectrum of TEAS at room temperature.

3100–2900 cm^{-1} with low intensity due to stronger polarized NH vibrations intensity. From the figure it is also shown that the absorption bands near 1400–1500 cm^{-1} are due to vibrations of $-\text{CH}_2$ and 1250–1391 cm^{-1} are due to $-\text{CH}_3$ of $\text{N}-\text{CH}_2-\text{CH}_3$ group of the trialkylammonium cation [27]. The other features in the finger print region of the spectrum (1500–500 cm^{-1}) are similar to the one observed by Mori et al. [25], Shmukler et al. [26] and Sayyar et al. [28] for the rest of trialkylamine side of the TEAS.

3.3 Measurement of the electrical conductivities of TEAS at different temperatures

Determining electrical conductivity of an electrolyte is the most important property. Electrical conductivities of TEAS containing 1.43 w/w % water were measured at 343, 353, 363, 373, 383 and 393 K and were 1.1, 1.4, 1.9, 2.5, 3.4 and 4.0 $\text{mS cm}^{-1} \pm 0.5\%$, respectively. The data was plotted as logarithm of the electrical conductivities versus reciprocal of temperatures ($\log \sigma$ vs. $1/T$) using Arrhenius kinetic equation (Eq. (1)),

$$\log \sigma = \log \sigma_0 - \frac{E_a}{2.303k_B T} \quad (1)$$

where σ is direct conductivity of TEAS, σ_0 is the conductivity of pre-exponential factor, E_a the activation energy, k_B the Boltzmann constant, 1.38×10^{-23} J/K and T the absolute temperature (K).

The $\log \sigma$ versus $1/T$ plot for TEAS is shown in the Figure 3 which is linear and follows Arrhenius kinetic model with correlation co-efficient 0.9959 giving a good statistical fit as observed also in our previous work for triethylammonium bisulphate (TEABS) [28].

It is noted that the E_a for TEABS sample (1.43 w/w % H_2O) calculated using the slope of the plot of Arrhenius equation was ~ 31.68 J/mol showing that the ions require lower energy for migration. With increase in temperature, the viscosity of the PIL also decreases which in turn increases the kinetic energy of the ions in the PIL and hence the electrostatic interactions between the opposite ions weaken. The result is an enhance ionic conductivities.

3.4 Electrochemical characterization of TEAS

EW of TEAS was determined using cyclic voltammetry. It is an important indicator of electrochemical stability of ionic liquids. It is the potential range within which the substance under study remain inert and can be determined from the difference of oxidation (anodic) and reduction (cathodic) potential limits. Identification of this potential range (EW) for given solvents and electrolytes is one of the most important characteristics in relevance to their use in electrochemical applications [29]. The protic ionic liquid, TEAS was, therefore, electrochemically characterized by measuring its potential window and various adsorptions and desorption reactions taking place in this media at Pt and Au working electrodes using cyclic

Figure 3: Arrhenius plot of temperature dependence of ionic conductivity of TEAS.

voltammetry. Figure 4 shows CVs recorded in N_2 saturated blank TEAS at Pt (A) and Au (B) disc electrodes versus Ag wire at 50 mV/s and at 333 K. Due to lack of electrochemical literature about TEAS, few trial base experiments were performed for measuring the positive (anodic) and negative (cathodic) potential limits at each electrode.

Figure 4: Cyclic voltammograms obtained at a 2 mm diameter. Pt disc (A) and Au disc (B) electrodes in N_2 purged TEAS at 333 K at a sweep rate of 50 mV s⁻¹. Inset of Figure 4(A) show CV portion range from 0.5 to 1.15 V and Figure 4(B) at Au from 0.9 to 1.7 V.

It is evident from Figure 4(A) that four oxidation processes, a_1 , a_2 , a_3 and a_4 are taking place during the positive going sweep in TEAS at Pt disc electrode with their corresponding reduction processes, c_1 , c_2 , c_3 and c_4 on the reverse sweep. The sharp increase in current at more positive potentials (a_4) in the PIL corresponds to the oxidation of the anion, $[\text{SO}_4]^{2-}$ (Eq. (2)). On reversing the scan direction after reaching anodic potential limit, the current (c_4) observed is due to the corresponding reduction of the species formed during the forward sweep (Eq. (3)).

Cathodic current c_1 corresponds to the reduction of the PIL cation; triethylammonium $[\text{TEA}]^+$ (Eq. (4)). This reduction results in the formation of triethylamine. This represents the cathodic potential limit of the PIL.

The bases formed by the reduction of the cations, as described above, remain in the vicinity of the electrode, and oxidize back to their respective cations during forward scan depicted by the steep oxidation current (a_1) in the CV and represented by Eq. (5).

A similar oxidative and reductive decomposition of the anion and cation of the PIL is observed at Au disc electrode denoted by a_3 and c_1 , respectively along with their reverse reduction and oxidation processes (c_4 and a_4). The potential windows at 333 K calculated for TEAS versus Ag wire were approximately 3.5 V at Pt and 3.8 V at Au.

In the CV obtained at Pt in the PIL (Figure 4(A)), an additional anodic current (labelled a_3) was observed when the potential of the electrode was scanned in positive direction which is not observed at Au electrode in the medium. This oxidative current we attribute to the bulk oxygen evolution followed by another oxidative wave that may be attributed to oxidation of the hydrogen sulphate ion, $[\text{HSO}_4]^-$ (Eq. (6)) left unreacted during preparation of the PIL, TEAS, as also seen a peak for it at chemical shift 6.415 ppm in the ^1H NMR (Figure 1). The H^+ ions formed remain localized to the electrode and are reduced on Pt by scanning the potential of the electrode in the negative direction at around 0.0 V (labelled as c_4) to form Pt-H adsorbed specie (Eq. (7)). Pt-H is oxidized again in positive going sweep after the oxidation of the base TEA.

The wave corresponding to oxidation currents (a_3) for oxygen evolution reaction cannot be seen in the CV obtained in TEAS at Au disc electrode. The oxidation wave of $[\text{HSO}_4]^-$ in positive going scan at Au is probably merged, due to high overpotential, with the oxidative decomposition current for $[\text{SO}_4]^{2-}$ ions. This is evident from the broad oxidative wave at potential beyond 2 V. Similarly the corresponding reduction of H obtained from $[\text{HSO}_4]^-$ oxidation occur at high overpotential and overlap with the reduction of the TEAS cation as can be seen from the broad reduction wave at potentials below -0.2 V. The inset of each of the Figure 4 shows the middle portion of the window CVs, at Pt (Figure 4(A)) from 0.5 to 1.15 V and at Au (Figure 4(B)) from 0.9 to 1.7 V that corresponds to surface oxidation of Pt and Au electrodes, respectively by trace water (1.43 w/w %) in the TEAS. As shown in the inset of Figure 4(A), an oxidation wave at an onset potential 0.95 V with peak potential at 1.02 V at Pt and at an 1.2 V having peak potential 1.5 V at Au electrodes versus Ag wire. These oxidation currents corresponds to adsorption and oxidation of trace water present in TEAS (1.43 w/w %) forming Pt-OH/PtO and AuO/Au₂O₃ at Pt and Au, respectively [30, 31].

If we scan the potential of the electrode further beyond the switching potentials, the current due to reductive and oxidative decomposition of the PIL will drastically increase so that all the ionic liquid decompose to another product. However, the window is not affected after further scans. The more readily oxides are formed on electrode surface due to oxidation of trace water of PILs the more window gets narrower. The amount of the formation of oxides increases and the window gets narrower with rise in water content of the PIL. This can cause decrease in the stability of the ionic liquid.

The corresponding reduction peak negative of 0.6 V at Pt and 1.2 V at Au in the PIL is also observed, which looks very small in the wide window voltammogram shown in Figure 4. However, in the narrower potential window, shown in the inset, a clear cathodic wave is seen. These respective reduction waves corresponds to the stripping of the adsorbed oxides from the surface of Pt and Au back forming clean Pt and Au surfaces. These oxides formed during positive potential sweeps and the residual oxides left of the electrodes surfaces could act as co-catalysts for the oxidation of liquid fuels such as hydrazine [30], methanol [32] and formic acid [1].

3.5 Variation of EW of TEAS with temperature

With the rise in temperature of an electrolyte, its EW decreases [26]. The effect of increasing temperature on the EW of TEAS was elucidated at Pt and Au electrodes and the CVs obtained at 333, 353 and 373 K are shown in the Figure 5(A) and (B),

Figure 5: Electrochemical windows (EWs) of triethylammonium sulfate (TEAS) at different temperatures at 50 mV/s at (A) Pt, (B) Au disc working electrode versus Ag wire reference electrode.

Inset of each figures shows the effect of temperature on the electrode-surface oxides formation and stripping regions.

respectively. Both the positive and negative potential limits are affected by changing temperature of the medium. It is evident from Figure 5, by rising temperature, the reduction peak at less negative potentials and the oxidation peak appears at less positive potentials decreasing separation between the two peaks that narrows down EW of the ionic liquid. This showing that with increase in temperature, the rate of oxidation and reduction processes increases and also the magnitude of both the oxidative and reductive currents increases as evident from the peaks heights. On Pt electrode the EW of TEAS at 333, 353 and 373 K are 3.5, 3.3 and 3.1 V, respectively. Similarly at Au electrode in TEAS the EW at 333, 353 and 373 K are 3.8, 3.1 and 2.9 V, respectively. At both the electrodes the net decrease in the EW is 0.5 V. The temperature increase also affects the currents and potentials of the electrode surface oxidation and reduction processes [30–32]. It is clear from the CVs in the inset of Figure 5(A) at Pt and Figure 5(B) at Au electrodes that as the temperature increases the onset of surface oxides formation on both the electrodes moves toward more negative potentials in the forward going scan. Also the oxides stripping off the electrodes happens earlier in the reversed going sweeps.

4 Conclusions

A protic ionic liquid, TEAS was successfully synthesized as ^1H NMR and FT-IR spectra confirm the proton transfer and its formation. The water content of the PIL was 1.43 w/w % measured by Karl-Fisher titration. The electrical conductivity of TEAS was high which further increase linearly with the increase in temperature and follows Arrhenius kinetic model. Electrochemical windows of TEAS was wider at Au electrode than at Pt electrode at all the temperatures studied and the windows decrease with rise in temperature from 333 to 373 K at both Pt and Au disc working electrodes. During positive potential scans, oxides were formed on Pt and Au electrodes surfaces due to water oxidation. The amount of oxides increases with increase in temperature as evident from the increased oxidation current and shifting of onset potential of oxide waves at both Pt and Au electrodes.

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