



Cyclic voltammetric studies of a new 1-pentylpyridazinium bromide ionic liquid and its determination in detergents and sea water samples



Ali F. Alghamdi

Department of Chemistry, Taibah University, 30002 Al-Madina Al-Munawara, Saudi Arabia

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ABSTRACT

The cyclic voltammetry technique was used to electrochemically analysis 1-pentylpyridazinium bromide (PPB) ionic liquid on hanging mercury drop electrode (HMDE) vs Ag/AgCl as reference electrode using different electrolyte buffers such as Britton-Robinson (B-R), phosphate, acetate and carbonate under changing values of pH. A sharp cyclic voltammogram was observed at $E_{ac} = -0.65$ V as reduction wave in case of using phosphate buffer pH 2.5. Other experimental conditions such as accumulation time and potential, sweep rate, voltage step, temperature, convection rate and area of working electrode surface were also evaluated. The cyclic voltammetric method has been explained an irreversibility of the reduction process and a characterize adsorption of analyte. The analytical performance of CV method for analysis of PPB was also evaluated in terms of studies of calibration curve, detection limit, recovery, stability and reproducibility. The calibration curve was studied over the range 1.0×10^{-5} to 5.0×10^{-4} mol L⁻¹, $r^2 = 0.997$ and $n = 13$ under the optimum parameters. The detection limit was calculated to become a 6.4×10^{-7} mol L⁻¹. Analyte was characterized a good stability within 180 min inside of a good reproducibility for eight voltammetric measurements, so the relative standard deviation (RSD%) was also reported 0.33%. The cyclic voltammetric applications were directly done for determination of PPB ionic liquid in the detergents and sea water samples.

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1. Introduction

Since the past two decades, ionic liquids (ILs) have attracted considerable attention as friendly environmental substitutes for volatile organic solvents due to the several unique properties such as negligible pressure, good stability, simple recyclability, no flammability, high ionic conductivity [1]. Due to these rarity properties, ionic liquids have been widely synthesized and analyzed as media for electrodeposition of metals [2,3], catalysis and biocatalysis [4,5], corrosion inhibition [6,7], food chemical science [8] and nuclear industry [9].

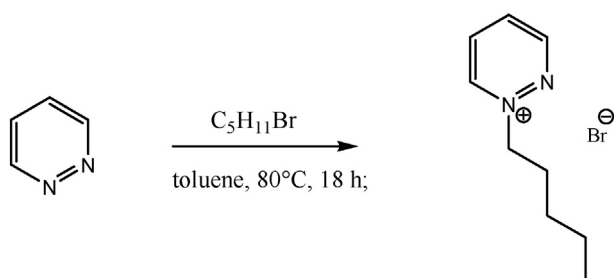
Cyclic voltammetry (CV) is an electrochemical technique which measures the current that develops in an electrochemical cell under conditions where voltage is in excess of that predicted by the Nernst equation. It is performed by scanning the potential of a working electrode, and measuring the resulting current. Cyclic voltammetry can be used to study qualitative information about electrochemical processes under some conditions, such as the presence of intermediates in redox reactions, the reversibility of reaction [10–14]. Cyclic voltammetry can also be used to evaluate the electron stoichiometry of a system, the diffusion coefficient of analyte, and the reduction potential, which can be

used as an identification device. Additionally, because concentration is proportional to current in the reversible, Nernstian approach, concentration of unknown solutions can be determined by generating a calibration curve of current vs concentration [15,16]. There are many published articles were described the using of cyclic voltammetric technique to determine organic, inorganic compounds and metals [17–25]. These present studies were performed to evaluate cyclic voltammetric assay of 1-pentylpyridazinium bromide (PPB) ionic liquid under optimized analytical conditions to develop sensitivity and selectivity of the used technique for determination of PPB in detergents and sea water samples. The analyte is a new ionic liquid and no analysis of this compound (PPB) using cyclic voltammetric technique was reported before.

1.1. Synthesis of 1-pentylpyridazinium bromide

To the solution of pyridazine (1 eq) in toluene, was added pentyl bromide (1.1 eq) at room temperature, followed by stirring at 80 °C for 18 h. The completion of the reaction was marked by the separation of oil from the initially obtained clear and homogenous mixture of pyridazine and pentyl bromide in toluene. The product was isolated by extraction to remove the unreacted starting materials and solvent. Subsequently, the pyridazinium ionic liquid was washed with ethyl

E-mail address: alifh2006@hotmail.com.



Scheme 1. Synthesis of 1-pentylpyridinium bromide (PPB).

acetate and dried at a reduced pressure to get rid of all the volatile organic compounds (Scheme 1).

1.2. Characterization of 1-pentylpyridinium bromide

^1H NMR (400 MHz, DMSO) δ : 0.97 (t, $J = 7.2$, 3H), 1.42 (quint, $J = 7.2$, 2H), 2.10 (quint, $J = 7.2$, 2H), 2.31 (quint, $J = 7.2$, 2H), 4.90 (t, $J = 7.2$, 2H), 8.55–8.56 (dd, $J = 1.2$, 1H), 8.61–8.63 (dd, $J = 1.2$, 1H), 9.53–9.54 (d, $J = 1.2$ 1H), 9.74–9.76 (d, $J = 1.2$ 1H); ^{13}C NMR (100 MHz, DMSO) δ : 15.2 (CH₃), 21.3 (CH₂), 32.7 (CH₂), 34.1 (CH₂), 68.1 (CH₂), 138.4 (C-4), 139.0 (C-5), 151.7 (C-6), 157.1 (C-3).

2. Experimental

2.1. Ionic liquid PPB

The new compound was synthesized and characterized by ^1H NMR and ^{13}C NMR, ^1H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were measured in CDCl₃ at room temperature. Chemical shifts (δ) were reported in ppm to a scale calibrated for tetramethylsilane (TMS), which is used as an internal standard.

2.2. Voltammetric analysis

Cyclic voltammetric studies were carried out by 797 AV computrace (Metrohm, Switzerland Made). Three electrodes system were applied in the current analysis that included hanging mercury drop electrode (HMDE) working electrode, Ag/AgCl reference electrode and Pt auxiliary electrode. The cyclic voltammograms were printed via a hp color laserjet CP1215 printer. For changing and getting the suitable pH values, Hanna instrument pH 211 (Romanian Made) was used. In addition, oxford adjustable micropipettes (Ireland) 100–1000 and 10–100 μL were used to inject analyte standard and studied solutions inside electrochemical cell.

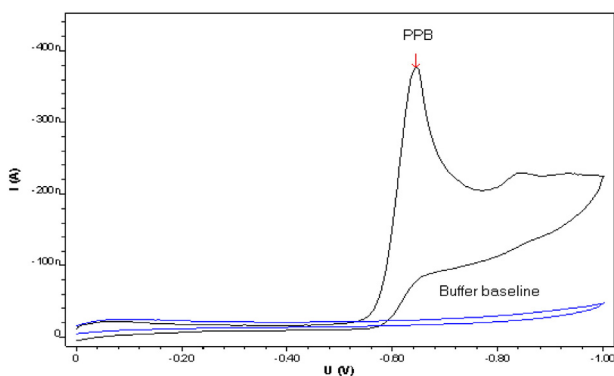
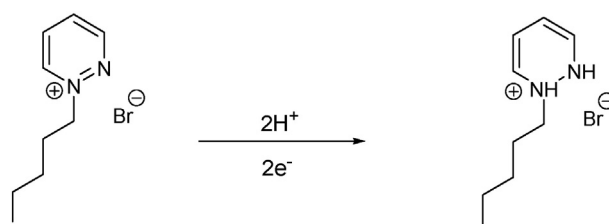


Fig. 1. Cyclic voltammogram of 1.0×10^{-4} mol L⁻¹ PPB in phosphate buffer pH 2.5, 50 mVs^{-1} scan rate and $-0.65 \text{ V E}_{1/2}$.



Scheme 2. Structure and proposed reduction voltammetric mechanism of PPB ionic liquid.

2.3. Chemical materials

A new ionic liquid PPB was obtained in a reagent grade as above section and dissolved in distilled water to be prepared 1×10^{-2} mol L⁻¹ as a stock solution. It stored in specialized place in the lab without light under the room temperature. The lower concentrations of standard solutions of PPB were prepared by diluting the stock solution by distilled H₂O. The supporting solutions such as Britton-Robinson (B-R), phosphate, acetate and carbonate buffers were prepared to be suitable the cyclic voltammetric determinations of PPB ionic liquid as in the references [26,27].

2.4. Cyclic voltammetric procedure

A 10 ml of supporting buffer under the suitable pH value was injected in clean and dry electrochemical cell. It was purged by a grade five nitrogen gas for 100 min with stirring. The standard solution of PPB was added according to a studied and wanted concentration. The reduction sweeps were monitored over the range 0.0 to -1.0 V under the optimum conditions such as accumulation time and potential ($10 \text{ s t}_{\text{acc}}$, $-0.1 \text{ V E}_{\text{acc}}$) and other parameters using above mentioning three electrodes system.

3. Results and discussion

The cyclic voltammetric behavior of the studied ionic liquid indicates that electroactive PPB was accumulated effectively onto HMDE surface vs Ag/AgCl reference electrode. The used method confirmed an irreversible nature of the cathodic reduction for 1.0×10^{-4} mol L⁻¹ of PPB in phosphate buffer pH 2.5 and 50 mVs^{-1} scan rate, as shown in Fig. 1. The resulting CV cathodic current was nearly due to the reduction reaction of the double bond ($-\text{N}=\text{N}-$) in the ring of 1-pentylpyridinium bromide compound to be obtained a signal bond that illustrated in the suggested mechanism (Scheme 2). Multi-cyclic voltammetry additionally was evaluated by analysis 1.0×10^{-4} mol L⁻¹ PPB in phosphate

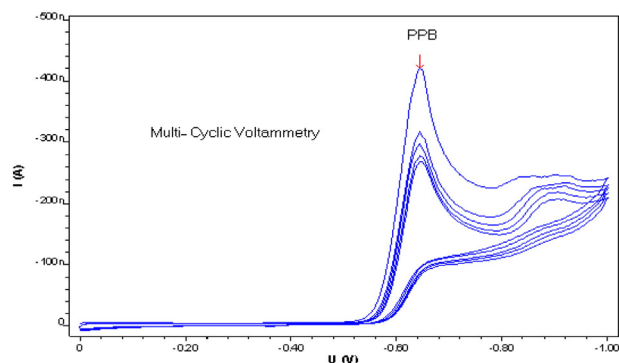


Fig. 2. Multi-cyclic voltammograms of 1.0×10^{-4} mol L⁻¹ PPB in phosphate buffer pH 2.5 and 50 mVs^{-1} scan rate (sweep (1) = 324, (2) = 242, (3) = 229, (4) = 215, (5) = 210 nA).

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