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CYCLIC VOLTAMMETRIC QUANTIFICATION OF THE AMINO COMPOUNDS WITH SPECIAL REFERENCE TO NAPHTHYL AMINE AT VARIOUS SOLID ELECTRODES

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ABSTRACT

The voltammetric behavior of some aromatic amino compound, such as alpha naphthyl amine and beta naphthyl amine was investigated and the method was developed for the simultaneous determination of these compounds. Adsorption of alpha naphthyl amine and beta naphthyl amine at solid electrode such as platinum and gold has been studied using cyclic voltammetry and linear sweep techniques. It was found that pH greatly influences characteristics of naphthyl amine. CV studies with platinum electrode at pH 4 gives second oxidation peak with no reduction peak whereas with gold electrode at pH 3 and 4 alpha naphthyl amine peak gives second oxidation peak with a cathodic peak. The effect of different parameters on the peaks response as supporting electrolyte, pH and scan rate were examined. Kinetic parameters were also calculated for the compounds.

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INTRODUCTION

Naphthylamines are substituted aromatic amines. According to the position of amine (-NH₂) group two isomers are possible viz alpha and beta. These compounds are stable, persistent and toxic. Naphthylamine is used in manufacturing of dyes, toning prints with cerium salts. Most of these compounds (i) are considered as poison by ingestion, subcutaneous, intraperitoneal and intramuscular routes, (ii) exhibit human mutagenic and carcinogenic potential, (iii) decompose to emit toxic fumes of and (iv) are potent uncouplers of oxidative- and photo phosphorylation. Between 1921 and 1951 case computed a number of bladder cancer deaths for men manufacturing azo dyes and compared this to the expected incidence of bladder cancer in England; four bladder cancer deaths were expected, whereas 127 deaths were found. Approximately 25% of all workers being exposed to aromatic amines, including β-naphthylamine and benzidine, developed bladder cancer. The workers, who were only exposed to benzidine, had further tumors (15%) than those being exposed to β -naphthylamine (50%).

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A few workers who distilled β - naphthylamine, all died of bladder cancer (Cartwright, 1983). α-naphthylamine was found to be a potent bladder carcinogen in dogs, but it was non-carcinogenic in rats and rabbits (Clayson and Garnerr, 1976). In spite of all those properties it is valuable feedstock for industries. The better electrochemical technique to study the oxidation of amino compound is the cyclic voltammetry. Anodic oxidation of 1- naphthylamine was studied in acetonitrile (Vettorazzi et al., 1981), in dimethylsulfoxide (Genies et al., 1987) NH₄F and HF medium by Genies et al (Marioli Juan, 1989). They used an optically transparent electrode for oxidation of α-naphthylamine. To see the temperature effect on oxidation of α-naphthylamine Juan et al (Vettorazzi et al., 1983) studied α- naphthylamine in methylene chloride. Electrochemical studies on α-naphthylamine and 1-aminoanthracene by ring-disk rotating electrode showed the existence of short lived primary products (Brittenhach and Heckner1971) Present work is devoted to the anodic oxidation of α-naphthylamine in aqueous medium. As found not much cyclic voltammetric work has been done on α-naphthylamine in a systematic way. So the aim of the study is to present the CV characteristics of α-naphthylamine in different aqueous mediums and at different working electrode

in different supporting electrolyte. Various kinetic parameters were also calculated.

Experimental

Preparation of Solutions: 1-0.1M aqueous solution of HCl, 0.1M H₂SO₄, 0.1M KCl, 0.1M KNO₃, 0.1M potassium hydrogen phthalate (pH4), 0.1M acetate buffer (pH 3 to 7) were prepared. The solutions of lower concentration for experiments were freshly prepared by appropriate dilution from stock solution prior to each run. The supporting electrolytes and buffers such as acetate buffer and potassium hydrogen phthalate were prepared using double distilled water. 2.2 Electrochemical Cell and 2.2 Instrumentation: A three electrode cell was used consisting of the Pt or Au as working electrode, a Ag/AgCl reference electrode and a Pt wire auxiliary electrode. Cyclic voltammograms were recorded with electrochemical analyzer CV-27, from Bioanalytical System Inc. (BAS), West Lafayette, USA in combination with a Series-100, Omnigraphic X-Y recorder, Houston Instruments, Austin (USA) and a cell stand C-1 (BAS, USA) served the purpose of electrochemical cell. The instrument is easy to use, versatile reliable and has broad applicability. Gold Electrode (AuE) and Platinum electrode supplied from Bioanalytical system Inc. (BAS), USA (Fig. 2.11). These electrodes are disk of highly pure electrode materials (viz. glassy carbon, gold or platinum) embedded in CTFE plastic rod.

Pretreatment of solid electrode

Surface was made by micropolishing before measurement using diamond lapping compound 1 μ m (Kemet, Kent, U.K.) and finally with 0.05 μ m α -alumina powder on a fabric pad attached to glass plates then thoroughly rinsed by deionized water. Further electrodes were held at initial potential for one minute before initiating every scan.

RESULTS AND DISCUSSION

Cyclic Voltammetric studies on α -naphthylamine at different electrode

• Effect of Electrolyte

Electrochemical studies on α -naphthylamine have been made at different working electrode (PtE and gold) and in different supporting electrolytes. Supporting electrolytes used for these studies were: KCl, KNO₃, H₂SO₄, HCl etc. For present CV studies initial potential is 0.0 V vs Ag / AgCl and final potential is 1.6 V vs Ag / AgCl. Cyclic voltammetric (CV) characteristics of α -naphthylamine in different electrolytes are included in Table 1.

• Effect of pH

Electrochemical oxidation of α -naphthylamine was also studied at different pH using acetate buffer (pH 3 to 7).

Table 1. Cyclic voltammetric characteristics of α -naphthylamine in different supporting electrolyte: Reference electrode: Ag/AgCl Concentration: 1mM Scan Rate: 100 mVs⁻¹

* Potassium Hydrogen Phthalate (pH4) # Acetate Buffer (pH 3)

Supporting electrolyte	First Oxidation Peak		Second Oxidation Peak		Reduction	
	Epa (V)	Ipa (μ A)	Epa (V)	Ipa (μ A)	Epc (V)	Ipc (μ A)
	<i>Platinum electrode</i>					
0.1M KCl	1.02	28	-	-	-	-
0.1M KNO ₃	1.01	26.5	-	-	-	-
0.1 M PHT [*]	1.04	8.5	-	-	-	-
0.2 M AB [#]	0.96	22	-	-	-	-
0.1M H ₂ SO ₄	0.84	18.5	-	-	-	-
0.1M HCl	0.95	24	-	-	-	-
	<i>Gold electrode</i>					
0.1M KCl	1.04	31	-	-	-	-
0.1M KNO ₃	1.09	29	-	-	-	-
0.1 M PHT [*]	1.03	21	-	-	-	-
0.2 M AB [#]	1.0	21.5	1.58	8.5	0.67	3.5
0.1M H ₂ SO ₄	1.1	25	1.46	17.5	0.97	-
0.1M HCl	1.25	40.5	-	-	-	-

Table 2. Cyclic Voltammetric characteristics of α -naphthylamine in acetate buffer at different pH: Reference electrode: Ag/AgCl Concentration: 1mM Scan Rate: 100 mVs⁻¹

pH	First Oxidation Peak		Second Oxidation Peak		Reduction	
	Epa (V)	Ipa (μ A)	Epa (V)	Ipa (μ A)	Epc (V)	Ipc (μ A)
	<i>Platinum electrode</i>					
3	1.05	19	-	-	0.35	7.5
4	0.96	22	1.54	11.5	-	-
5	0.75	11	-	-	-	-
6	0.70	12.5	-	-	-	-
7	0.70	17	-	-	-	-
	<i>Gold electrode</i>					
3	1.15	12.5	1.65	12.5	0.95	5
4	1.0	21.5	1.58	8.5	0.67	3.5
5	0.75	12.5	-	-	-	-
6	0.74	11	-	-	-	-
7	0.67	16	-	-	-	-

CV characteristics are shown in CV datas compiled in Table 2. Some notable features of these studies are shown here.

Cyclic voltammograms with well-defined peaks were obtained in all the electrolytes used.

- When anodic scan extended beyond +1.4 V which was possible with gold electrode (only in H₂SO₄, acetate buffer) and with PtE (in acetate buffer) second oxidation signal could also be recorded and during cathodic scan one cathodic peak corresponding to second oxidation peak was observed (only at gold electrode.). This was confirmed by reversing the scan at the end of first peak, when no second peak was observed.
- A well- defined irreversible oxidation peak is obtained in all the pH ranging from 3 to 7.
- pH greatly influences the characteristics of cyclic voltammograms. CV studies with PtE at pH 4 gives second oxidation peak with no reduction peak; whereas with gold electrode at pH 3 and 4 α -naphthylamine gives second oxidation peak with a cathodic peak.
- Peak potential shift of the main oxidation peak was 350 mV and 480 mV towards negative potential for a 4 unit increase in pH at 100 mVs⁻¹ at Pt and gold electrode respectively.
- Peak height increases with increase in pH till 4 and then it decreases, after pH 6 it again incrAt low pH or in acidic medium (pH 3 & 4) second oxidation peak is obtained followed by first oxidation peak with gold electrode. This second oxidation peak on reversal of scan gives cathodic peak; but as the pH value increases these two peaks disappear. This shows that oxidation of α -naphthylamine is pH dependent.

α -naphthylamine is widely used in dye and tanning industries. Looking to the carcinogenic effects of the compound mentioned in above paragraph, the use of such chemicals in dyes industries should be restricted /banned.

Various Industries are still using such dyes even after the ban on use of all such dyes by various agencies such as NIOSH, OSHA, ECDIN etc. To prevent the use of such dyes, the detection of these carcinogenic chemicals in dyes effluent can be determined in an eco friendly manner using the present study. So the present study can help a lot in very low order detection and determination of such carcinogenic compounds.

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