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Electro Analytical Studies on Ethoxylation of O-Nitro Phenol

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Abstract: Ortho-Nitrophenol (1-hydroxy-2-nitrobenzene), a yellow crystalline solid (m.p. 46° C). It is a precursor to the rice herbicide fluorodifen and the pesticide parathion. The literature on various chemical reactions of o-Nitrophenol is scattered. Only a few works have been done on electrochemical studies of o-Nitrophenol. This work is concentrated on the cyclic voltammetric studies of o-Nitrophenol. Cyclic voltammograms were recorded by varying pH, working electrode and scan rate. Multiple scan studies have also been done. The system showed variations in the working potentials (anodic peak voltage) while the reaction conditions such as pH, working electrode, are changed. These studies also revealed that the ethoxylation of o-Nitrophenol is diffusion controlled at all mentioned above conditions. It is also found out that o-Nitrophenol is not forming polymers on the working electrode.

Key words: Cyclic voltammetry, o-Nitrophenol, working electrode, ethoxylation, electro analysis.

1. INTRODUCTION

O-Nitrophenol is a precursor to the rice herbicide fluorodifen and the pesticide parathion.¹⁻² Many chemical reactions of O-Nitrophenol have been studied extensively. Many electrochemical studies of O-Nitrophenol such as hydroxylation³, halogenations⁴, cyanation⁵ and acetoxylation⁶ have been reported. Only a few studies on ethoxylation of O-Nitrophenol are found in the literature. Recently the electrochemical analytical studies on methoxylation of various mono substituted aromatic compounds have been examined and reviewed.⁷⁻⁸

In this present work platinum and glassy carbon electrodes are taken as working electrodes. The electrode potentials of working electrodes are found out by taking Ag/AgCl electrode as reference. The ethoxylation of O-Nitrophenol was carried out in ethyl alcohol medium. KOH, KCl, H_2SO_4 are used as supporting electrolytes in alkaline, weakly acidic and strongly acidic conditions respectively. The work is carried out to predict the anodic peak potentials at different pH media, to find out whether the reaction is diffusion or adsorption controlled and to ascertaining the formation of any polymer films on the surface of the working electrode.⁹

2. MATERIALS AND METHOD

2.1 APPARATUS

Voltammogramms are recorded with potentiostat CH 10 (Sinsil international) interfaced to 663 VA stand (Metrohm) and SyncMaster B1930 computer. A three electrode configuration was used with platinum / glassy carbon electrode as the working electrode, a silver- silver chloride reference electrode and a platinum electrode wire as the auxiliary electrode.¹⁰ The working electrode is pretreated by polishing it with an alumina – water slurry followed by washing in an ultrasonic path.

2.2 REAGENTS AND SOLUTIONS

All reagents are of analytical grade and ultra pure water is used throughout 0.001 M O-Nitrophenol, 1M $H_2SO_4/KOH/$ KCl, 0.5M Ethanol are prepared freshly. The pH of the different reaction mixtures measured with pen type pH meter. The solutions are stored in a light protected cool location.

2.3 METHODOLOGY

The three electrode system with platinum/glassy carbon electrode as the working electrode, platinum

wire as the auxiliary electrode and Ag/AgCl electrode as the reference electrode is constructed in an undivided cell. In order to change the pH of the system 1M solutions of H_2SO_4 /KOH/ KCl were taken. These solutions are also working as the sources of supporting electrolytes. Then the cylclic voltammogram is recorded with different scan rates, different working electrodes and different pH conditions. To arrive at an idea about the polymerization of O-Nitrophenol on working electrode multiple scan cyclic voltammogram is also recorded.

<u>3. EXPERIMENTAL METHODS</u>

3.1 CYLIC VOLAMMOGRAMS OF O-NITROPHENOL AT DIFFERENT PH VALUES

O-NITROPHENOL+KOH+ETHANOL at pH 13

O-NITROPHENOL+KCL+ETHANOL at pH 6



| S.No | SUBSTRATE | pH | OXIDATION PEAKS (v) |
|------|-------------------------------|----|---------------------|
| 1 | o-nitrophenol +ethanol+KOH | 13 | -0.5 0.7 |
| 2 | o-nitrophenol +ethanol+Kcl | 6 | 1.3, -0.3 |

From the cyclic voltammograms the anodic oxidation potentials are predicted and tabulated

3.2 CYCLIC VOLTAMMETRY SCAN RATE VARIATIONS (o-nitrophenol +Ethanol + KOH)

o-nitrophenol: 0.001 M [alcohol]: 0.1M Anode: Pt Cathode: Pt at pH: 13

| | Scan rate (mV/s) | | | | |
|------------|------------------|--------|--------|--------|--------|
| Parameters | 100 | 200 | 300 | 400 | 500 |
| 1/2 | 10 | 14.14 | 17.32 | 20 | 22.36 |
| Ip (µA) | 110.3 | 167.3 | 220.4 | 260.4 | 299.8 |
| Ep (V) | 1.467 | 1.1834 | 1.2145 | 1.2354 | 1.2563 |



o-nitrophenol: 0.001 M [alcohol]: 0.1M Anode: Pt Cathode: Pt pH: 6

| | Scan rate (mV/s) | | | | |
|----------------|------------------|--------|--------|--------|--------|
| Parameters | 100 | 200 | 300 | 400 | 500 |
| 1/2 | 10 | 14.14 | 17.32 | 20 | 22.32 |
| Ιр (μA) | 75.6 | 120.3 | 150.4 | 175.4 | 204.2 |
| Ep (V) | 1.5106 | 1.5696 | 1.6402 | 1.6752 | 1.7165 |



3.3 ELECTRODE VARIATION STUDIES

The cyclic voltammograms are also recorded by replacing pt electrode by glassy carbon as a working electrode.



The following are the anodic and cathodic peaks potential observed by various working electrode

| S.NO | Substrate | working electrode (glassy carbon) | |
|------|----------------------------|-----------------------------------|------------------------|
| | | oxidation peaks ep (mv) | oxidation peaks ep(µa) |
| 1 | o-nitrophenol +ethanol+KOH | -0.0637 | 7.4803 |
| 2 | o-nitrophenol +ethanol+KCl | 0.8094 | -0.8094 |

3.4 MULTIPLE SCAN STUDIES

For a system capable of multiple scan technique, the potential sweep at a specific scan rate namely 100 mV/sec is repeatedly performed for 10 segments through the same solution, without forming polymer coats, the potential sweep is responsible for the first segment by way of initiating polymerization.

As the electrode get coated with the polymer of conducting nature, in the present study the cyclic voltammograms with such multiple scans for onitrophenol in aqueous solution are studied.



It is clearly understood that no polymeric coatings seem to have occurred in the systems. This decision has been derived from the observation that the peak current fluctuations at the respective peak potential do appear at multiple scan rates subsequent sweeps cannot show any peak current.

During the reaction at an electrode surface, material is depleted and a concentration gradient is set up. Reactant from the bulk of solution then diffused towards the electrode surface in response to the gradient. In a similar manner, products of the electrode reaction diffuse away from the electrode.

4. CONCLUSION

From the electroanalytic studies on aromatic ethoxylation of o-nitrophenol, the following conclusions are drawn.

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- The possible anodic oxidation potentials for the electro chemical ethoxylation of o-nitrophenol at Pt and Graphite are predicted.
- By changing the electrodes, the change in the oxidation potentials are also found out.
- By changing the pH of the reaction medium, the favorable pH for the ethoxylation of onitrophenol is predicted.
- By the scan rate variation studies it is also observed that the ethoxylation of o-nitrophenol is diffusion controlled and not adsorption controlled.
- The multiple scan rate studies of ethoxylation of o-nitrophenol revealed that no formation of polymer coating during the electrolysis.
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