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CYCLIC VOLTAMMETRIC STUDY OF CuO IN AQUEOUS AND NONAQUEOUS SOLVENTS

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ABSTRACT

A series of cyclic voltammograms were conducted by varying the sweep potential between -1 and +1.2 V of copper oxide (CuO) mixed with graphite paste. In the first anodic cycle one anodic peak was observed and in the reverse cycle two cathodic peaks appear. The peaks increase in height after consequent cycles. It was observed that in different solvent systems these trends are similar with slight changes in the potential and current density values. The above result indicates the reaction to proceed through a two electrons process. Various parameters like scan rate, scan reversal, concentration, pH, etc. were studied in both aqueous and non-aqueous solvent systems. The voltammograms obtained indicate the quasi reversible nature of copper oxide.

Keywords: Cyclic Voltammetry, CuO, DMSO, Nonaqueous solvents.

1. INTRODUCTION

Cyclic Voltammetry (CV) is the most versatile electrochemical techniques used in the field of electrochemistry, inorganic chemistry, organic chemistry and biochemistry [1-6]. It is perhaps the most effective technique available for the mechanistic study of redox systems [7-11]. it enables the electrode potential to be rapidly scanned in search of redox couples. It would be largely used to determine half-wave potentials of redox systems. It also gives information about the relative stabilities of the oxidised and reduced forms. It also provides the number of electrons involved in the reaction. Sometimes CV reveals the presence of new unsuspected compounds. Oxidised copper minerals cannot be effectively separated from carbonate gangue minerals by floatation and direct acid leaching is not economical because of excessive acid consumption. Although the recovery of copper from oxidised copper ores by ammonia or cyanide leaching is technically feasible, problems are often encountered in leachant generation. The main purpose of the leaching solvent chosen for hydrometallurgical processes to dissolve the desired mineral should be selective and fast. In the recent times a number of non-aqueous solvents have found increasing use as a solvent in electrochemical studies. There are various non-aqueous solvents like Dimethyl sulfoxide (DMSO), Dimethyl formamide (DMF), acetonitrile (ACN), etc.; which are used in the study of electrochemical techniques. Among the various non-aqueous solvents used DMSO finds a wide spread use in organic chemistry, biochemistry, industry and medicine [12]. Certain ions and gases which are difficult to estimate in other solvents can be determined in DMSO by polarograms and voltammograms. In present study the Cyclic Voltammogram of Copper oxide with graphic paste was recorded.

2. MATERIAL AND METHODS

All chemicals used throughout these studies were either of AR grade or were purified using known techniques [13]. CuO from S.D. Fine chemicals and were used as received. DMSO was of AR grade and was purified by keeping in 3A° molecular sieves to absorb water [14]. AR grade TBAP were obtained from Fluka and was oven dried for an hour at 100°C before the experiment. KCl was AR grade and was used as such. Sodium sulphite, Iodine, EDTA of AR grade were used and were obtained from S. D. Fine Chemicals. Graphite and paraffin oil used was of reagent grade. Nitrogen gas was obtained from Industrial Oxygen Ltd. with 99% purity. Sulfur dioxide gas was supplied by INOX Ltd. in small volume capacity and was used directly.

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2. MATERIAL AND METHODS

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2.1. Electrode systems

Electrochemical studies were conducted using a five necked round bottom flask. Electrochemical measurements were carried out using a three electrode system *i.e.*, working electrode (WE), an auxillary or counter electrode (CE) and a reference electrode (RE) [15,16]. The WE was made up of graphite paste, platinum wire served as a CE and the RE electrode used was SCE for aqueous solution and a silver wire which served as a quasi reference electrode (QRE) in non-aqueous solution. The solution was purged for about 1 hour before each experiment with pure nitrogen gas to remove the dissolved oxygen gas. The studies were performed in the inert nitrogen atmosphere which was maintained during the run of the experiment. The QRE was placed very near to the WE to eliminate the i_R potential drop in the solution. The measurements were taken in an unstirred solution at room temperature.

2.2. Estimation of copper

Copper was estimated by EDTA method [17]. About 200 mg of copper oxide was taken in a beaker and then 2 ml of conc. HNO_3, 2 ml of conc. H_2SO_4 and about 1-2 drops of 70% perchloric acid was added in it. The solution was heated strongly to dryness. The decomposed sample was dissolved in 5 ml of 2N HCl, then 10 ml of conc. ammonia solution was added. The resulting solution was titrated against 0.01 M EDTA solution using Fast Sulphon Black F as an indicator. The colour changed from blue to pale green near the endpoint. The metal % was calculated as:

1000 ml of 1M EDTA = 63.54 gm of copper.

2.3. Determination of Sulfur dioxide concentration

In a conical flask known volume of standard 0.1 N iodine solution (10-25 ml) depending upon the concentration of sulfur dioxide in the solution, was taken and diluted with distilled water to about 150 ml and acidified with 5 ml of 2 N HCl [18]. Then the known volume of aqueous sulfur dioxide solution was taken in a pipette was slowly added to the solution in the conical flask and the excess iodine was immediately titrated against standard 0.1 N sodium thiosulfate solution, using starch as an indicator. The total volume of standard iodine solution taken minus the burette reading gave the amount of iodine which has reacted with aqueous sulfur dioxide.

$SO_3^{-2} + I_2 + H_2O = SO_4^{-2} + 2H^+ + 2I^-$

$HSO_3^- + I_2 + H_2O = SO_4^{-2} + 3H^+ + 2I^-$

It follows that

1 ml 0.1 N I_2 = 0.0032 gm SO_2

In case of determination of sulfur dioxide in DMSO, due to high concentration of sulfur dioxide, very low volume of the solvent mixture *i.e.*; 0.1 ml was taken for iodometric analysis.

2.4. pH Meter

The pH of the solution was measured with pH-Elico instrument. The instrument was standardized using buffer tablets before each experiment.

2.5. Potentiostat

Electrochemical measurements were performed using a E.G & G Princeton Applied Research Corporation 273 Potentiostat/Galvanostat model. M270 electrochemical software was used to control the instrument. The instrument was hooked to a 386 computer along with a printer.

3. RESULTS AND DISCUSSION

3.1. Cyclic voltammetry of CuO in water sodium sulfite system

The cyclic voltammetric measurements were carried out to study the anodic/cathodic peak potentials and currents of CuO in water-sodium sulfite system. They were studied with respect to scan rate, concentration, potential scan reversal, pH, etc.

The cyclic voltammetric measurements were taken in a solution purged with nitrogen gas. CuO was mixed with graphite in a fixed ratio and it served as the working electrode. 0.1M KCl was used as the supporting electrolyte. SCE served as the reference electrode and platinum wire as an auxiliary electrode. Cyclic voltammograms were measured at fixed scan rate of 20 mV/s. Cyclic voltammetric measurements were made at different pH values of 8.8, 3.0, 2.2, 1.8 and 1.5. They are shown in fig. 1 & 2. Cyclic voltammogram were obtained both upto 1.0 V and 1.3 V at a particular pH values.

In the first cycle, no anodic peak appears, but in the reverse scan one cathodic hump appears. In the consequent cycle, an anodic peak appears on the positive scan and in the negative scan two cathodic peak appears. As the number of cycles is increases, the peak height increases. In case of multicyclic voltammograms, the anodic peak potentials shifts towards more positive potential and conversely the cathodic peak moved towards more negative potential. The shift observed has been explained earlier. As the number of cycles is

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increased, the two anodic peaks merges to form a broad peak. Similarly, the two cathodic peak merges to form a broad cathodic peak. This is due to the increase in the peak height which merges with the smaller anodic/cathodic peaks.

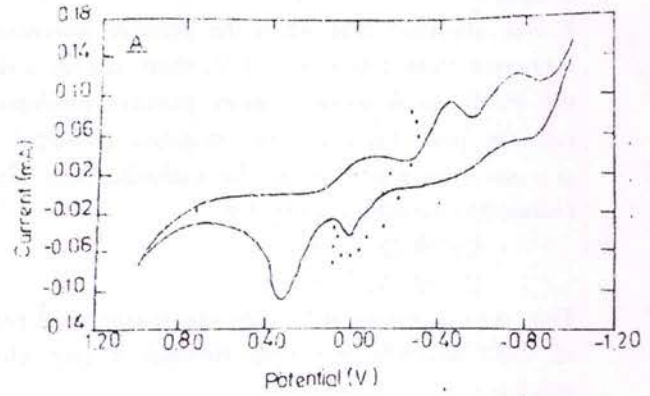

Fig. 1: Cyclic Voltammogram of CuO in Water-Sodium sulphite system at pH 2.2 (upto 1.0 V)

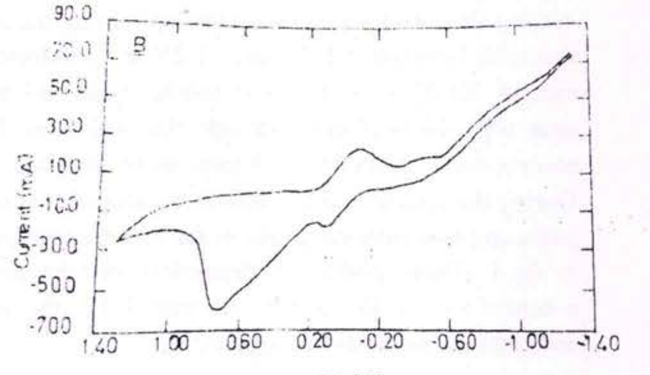

Fig. 2: Cyclic Voltammogram of CuO in Water-Sodium sulphite system at pH1.8 (upto 1.3 V)

When the potential was increased from 1.0 V to 1.3 V, the anodic and cathodic peak height increases comparatively. When the positive potential was increased beyond 1.3 V, initially the anodic and cathodic peaks gets separated and gradually the cyclic voltammograms gets distorted.

In order to determine the reversibility of the anodic and the cathodic peaks, cyclic voltammograms were run in different potential range. The potential range was varied both in the positive direction as well as in the negative direction. When we measured the cyclic voltammetry from 0.0 V to +1.0 V, we observed that there was no cathodic peak. Similarly when we measured in the negative direction i.e., from 0.0 V to -1.0 V, there is no appearance of the anodic peak. We can thus deduce that the cathodic peak is due to the anodic peak which appears when we run the cyclic voltammograms from -1.0 V to +1.0 V, which was not observed in either the positive scan or the negative scan. The two anodic peak appears around 0.2 V and 0.3 V and the cathodic peak appears around 0.0 V. The peaks at pH 2.2 are the sharpest. At pH 1.8, the second anodic peak shifts around 0.7 V.

It was observed that when the positive potential was increased from 1.0 V to 1.3 V, there occurs a shift of the anodic peak towards more positive potential and cathodic peak towards more negative potential. Thus we can conclude that in the cathodic scan CuO is reduced to metallic copper, i.e.;

$$Cu^{0} \rightarrow Cu^{+1} + e^{-}$$
$$Cu^{1} \rightarrow Cu^{+2} + e^{-}$$

Thus we can interpret that the electrochemical reaction of CuO basically proceeds through a two electron process.

3.2. Cyclic Voltammetry of CuO in DMSO

CuO was mixed with graphite paste and the cyclic voltammograms were measured in DMSO solution. The cyclic voltammetry was measured by varying the sweep potential between +1.2V and -1.2V at a constant scan rate of 20mV/s. In the first anodic cycle, no anodic peak was observed but a straight line was seen. In the reverse cycle, two cathodic humps were observed.

During the second anodic cycle there appear two anodic peaks and two cathodic peaks in the reverse scan as seen in fig.3. These peaks are dependent on the positive potential i.e., 1.2V. If it is less than 1.2V, the anodic and cathodic peaks do not appear.

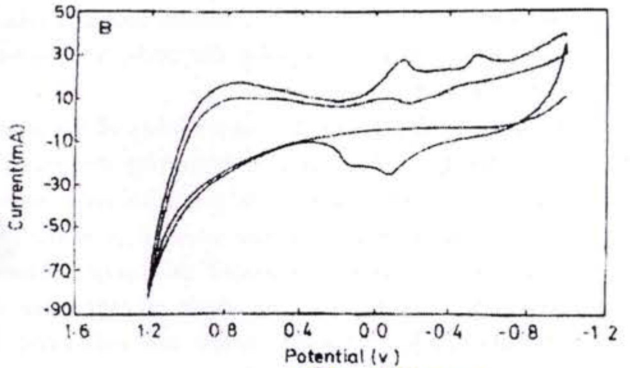

Fig. 3: Cyclic Voltammogram of CuO in Dimethyl Sulfoxide (2^{nd} cycle)

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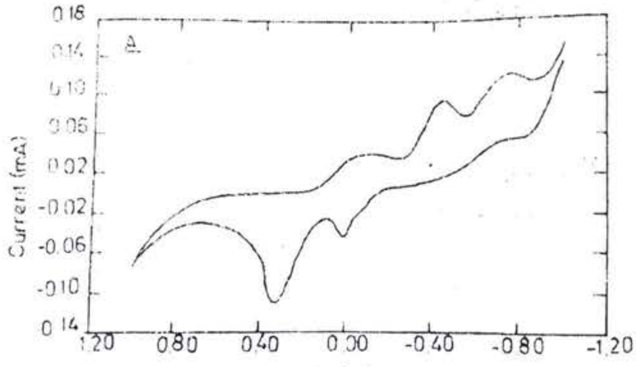

Fig. 1: Cyclic Voltammogram of CuO in Water-Sodium sulphite system at pH 2.2 (upto 1.0 V)

Fig. 2: Cyclic Voltammogram of CuO in Water-Sodium sulphite system at pH1.8 (upto 1.3 V)

When the potential was increased from 1.0 V to 1.3 V, the anodic and cathodic peak height increases comparatively. When the positive potential was increased beyond 1.3 V, initially the anodic and cathodic peaks gets separated and gradually the cyclic voltammograms gets distorted.

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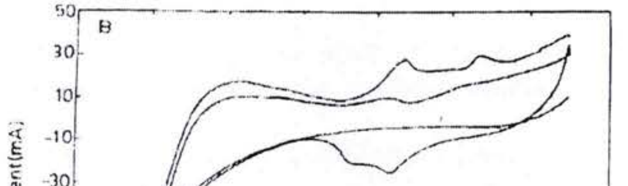

Fig. 3: Cyclic Voltammogram of CuO in Dimethyl Sulfoxide (2nd cycle)

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If the positive potential is increased to 1.3V, the anodic and cathodic peak heights increases comparatively. As the number of cycles is increased and the cyclic voltammetry is run for a long time, the two anodic peaks merges to form a broad anodic peak as shown in fig.4. This may be due to increase in the peak height which merges with the smaller anodic peak.

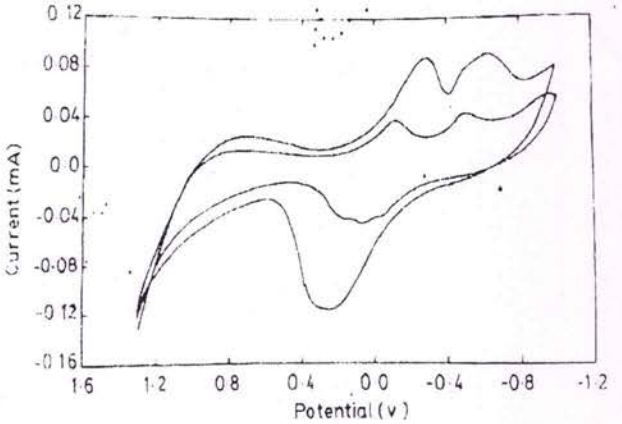

Fig. 4: Cyclic Voltammogram of CuO in Dimethyl Sulfoxide (From positive to negative scan)

As the number of cycles is increased, the anodic peak shifted towards a more negative potential. There is also an increase in peak height. If we start the cyclic voltammograms from positive potential say 1.3V, the cathodic peak appears as usual, but there are two anodic peaks in the first cycle itself as compared to when we start from negative potential where there is no anodic peak in the first cycle. The anodic peak potential and current values are shown in table 1 and the cathodic peak potential and current values are shown in table 2.

In order to find out the nature of oxidation and reduction peaks, we have carried out several short scans. The potential range was varied both in the positive direction as well as in the negative direction. When we measure the cyclic voltammetry from 0.0 V to +1.0 V, we observe that there was no cathodic peak. Similarly when we measured in the negative direction i.e., from 0.0 V to -1.0 V, there is no appearance of the anodic peak. We can thus deduce that the cathodic peak is due to the Anodic peak which appears when we run the cyclic voltammograms from -1.0 V to +1.0 V, which was not observed in either the positive scan or the negative scan. The anodic peak appears around 0.2V.

Table 1: Peak Anodic Potential and current values of CuO in DMSO

Cycle	I_{pa1}	E_{pa1}	I_{pa2}	E_{pa2}
No.	10^{-6} A	(V)	10^{-6} A	(V)
1	--	--	--	--
2	-25.90	-0.08	-22.10	0.11
3	-21.80	-0.08	-23.20	0.12
4	-105.3	0.20	-89.50	0.32
5	--	--	-109.3	0.23
6	--	--	-141.0	0.33

Table 2: Peak Cathodic Potential and current values of CuO in DMSO

Cycle	I_{pc1}	E_{pc1}	I_{pc2}	E_{pc2}
No.	10^{-6} A	(V)	10^{-6} A	(V)
1	6.57	-0.06	--	--
2	27.7	-0.15	30.4	-0.55
3	37.9	-0.16	41.8	-0.55
4	88.2	-0.29	91.6	-0.63
5	90.1	-0.29	92.6	-0.64
6	115.4	-0.36	124.8	-0.72

Therefore we may conclude that the cathodic peak arises from the electroreduction of CuO(s) to copper metal. Thus

$$Cu^{+2} + 2e^{-} \rightarrow Cu^{0}$$

Similarly the anodic peak is due to oxidation of the metallic copper to Cu^{+2} ion. Hence

$$Cu^{0} \rightarrow Cu^{+2} + 2e^{-}$$

Based on the results obtained above, the electrochemical reaction is said to proceed through a two electron process.

4. CONCLUSION

Based on the results obtained, it can be concluded that the electrochemical reaction of copper oxides proceeds through a two electron process. The voltammograms obtained indicate the quasi reversible nature of copper oxide.

Conflict of Interest

It is hoped that these investigations would contribute significantly in the fundamental understanding of the electrochemical leaching behaviour of copper oxide in various solvent systems.

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