ELECTROCHEMICAL REDUCTION OF O-NITRO-ANILINE AT AMALGAMATED COPPER AND AMALGAMATED LEAD ELECTRODES

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EXPERIMENTAL

The details are the same as in the preceeding paper [1].

RESULTS AND DISCUSSION

Polarization curves obtained by plotting potential vs. log c.d. are given in Fig 1. These were used to obtain the current density range in which electrochemical reduction in a divided cell was carried out. Percentage yield was determined at different current densities to choose the optimum value.

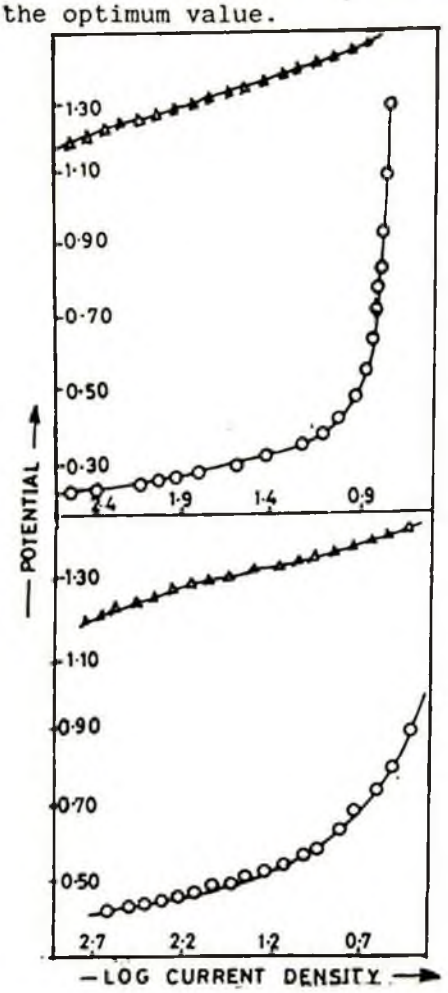


Fig. 1 Cathodic polarization curve
a) Amalgamated copper electrode
b) Amalgamated lead electrode

From Table 1 it can be easily seen that favourable current density for the reduction under study are 0.0740 amp/cm^2 for amalgamated copper and 0.0757 amp/cm^2 for amalgamated lead cathode. Using these values of current densities for each of the electrodes, other parameters such as temperature, depolarizer ratio and medium concentration were varied one at a time to obtain the desired optimum conditions for maximum yield, and the results are given in Table 2.

TABLE 1

EFFECT OF CURRENT DENSITY

Temperature 30 (±1)° C; Depolarizer Ratio 1:40, Medium concentration 10% H_2SO_4

C.D. Amp/cm^2	Yield %	C.D. Amp/cm^2	Yield %	C.D. Amp/cm^2	Yield %
Amalgamated Copper Cathode					
0.087	70	0.0740	80	0.1312	75
Amalgamated Lead Cathode					
0.0407	75	0.0757	80	0.0986	72

TABLE 2

EFFECT OF EXPERIMENTAL PARAMETERS ON YIELD

Depolarizer ratio 1:40 Medium 10% H_2SO_4		Temp. 45°± C Medium 10% H_2SO_4		Temp. 45°± C Depol. ratio 1:40	
Temp.	% I	Depol.	% I	% H_2SO_4	% I
Amalgamated Cu cathode; C.D = 0.0740 Amp/cm^2					
30	80	1:50	85	10	85
45	85	1:40	85	15	88
60	80	1:30	80	20	88
Amalgamated Lead Cathodes; C.D. 0.0757 Amp/cm^2					
30	75	1:50	86	10	86
45	80	1:40	86	15	88
60	80	1:30	80	20	88

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TABLE 3

EFFECT OF ADDITIVES

Temperature 45 (± 1)° C, Depolarizer Ratio 1:40, Medium 15% H_2SO_4

Cathode	Current density Amp/cm^2	Additive	Yield %
Amalgamated Copper	0.0740	Titanium sulphate	92
	"	Vanadium sulphate	92
Amalgamated Lead	0.0757	Titanium sulphate	94
	"	Vanadium sulphate	94

Some addition agents having different oxidation states (Titanium & Vanadium) are reported to increase the yield [2,3]. When such additives are added, higher valency ions are reduced at the cathode (e.g. Ti (IV)------→ Ti (III) and the lower oxidation state acts as a strong reductant thus increasing the yield Reduced species (Ti III) gets oxidised to (Ti IV) which is again reduced at cathode. Hence a small amount of such additive is sufficient. Effect of titanium sulphate and vanadium sulphate additives on the yield of o-phenylenediamine at optimum experimental contions was therefore studied and results are given in Table 3.

REFERENCES

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