

Electroreduction of 1-nitronaphthalene to 1-naphthylamine using TiO₂/Ti composite electrode

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The preparation of α -naphthylamine, has been investigated by electroreduction of α -nitronaphthalene. The potentiality of TiO₂/Ti composite electrode has been explored for the above preparation. Different experimental parameters such as current density, acid concentration and temperature have been optimised. The formation of product is confirmed by the preparation of derivative of formic acid and the melting point measurement has confirmed this.

Key words: Ti/TiO₂ electrode, electroreduction, 1-naphthylamine

INTRODUCTION

Titanous-titanic redox system has been used successfully for the reduction of more than 17 nitro compounds to their respective amino compounds in sulphuric acid medium and the yield reported is more than 90% for almost all compounds [1]. The same technique when used for the reduction of 1-nitronaphthalene to 1-naphthylamine, the yield was around 85% [2]. TiO₂ coated over titanium has been used earlier for the reduction of nitro compounds to their respective amino compounds [3-5]. The same electrode was used for the reduction of 1-nitronaphthalene to 1-naphthylamine also.

EXPERIMENTAL

The ceramic TiO₂/Ti electrode was prepared as reported [3-5]. The reduction of 1-nitronaphthalene to 1-naphthylamine at the TiO₂/Ti electrode was carried out at different acid strengths, current densities and temperatures

and the conditions for maximum yield of amine were optimised.

RESULTS AND DISCUSSION

The results are shown in Table I. It is evident from the results that the yield and current efficiency increase with current density (Expts. 1 to 3 in Table I), as well as with the temperature (Expts. 4 to 6 in Table I). It is also evident that the yield and C.E. decrease with the acid concentration (Expts. 2,6 and 7.)

CONCLUSION

Summing up the results from Table I, it is evident that TiO₂/Ti electrode can be used in a divided cell under stationary conditions using it as cathode and lead as anode with 30% H₂SO₄ (V/V) as the electrolyte. At temperatures between 343-353K and current density of 8A.dm⁻², the yield and current efficiency of 1-naphthylamine are 92.4% and 84.5% respectively.

TABLE-I: Electroreduction of 1-nitronaphthalene(NN) to 1-naphthylamine(NA) using TiO₂/Ti electrode

Area of the electrode 0.37dm²

Current (A)	C.D. (A.dm ⁻²)	Electrolyte strength (H ₂ SO ₄ V/V) (%)	Temperature (K)	Wt. of NN added (g)	Wt. of NA estimated (g)	Yield (%)	C.E. (%)
1	2.7	30	343-353	5.0	4.6	76.2	71.3
2	5.4	30	343-353	10.5	10.4	82.0	80.3
3	8.1	30	343-353	12.0	11.7	92.3	84.5
2	5.4	30	323-333	7.8	6.5	68.5	60.1
2	5.4	30	333-343	8.1	7.5	76.0	69.2
2	5.4	20	343-353	7.0	5.2	61.3	48.3
2	5.4	10	343-353	7.0	4.6	56.2	43.0

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