

ELECTROCHEMICAL REACTOR FOR PREPARATION OF MONOCHLOROTOLUENES

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Electrochemical preparation of monochlorotoluenes from toluene using divided/undivided electrochemical reactors in aqueous hydrochloric acid has been investigated under different experimental conditions to maximise the product yield. The process conditions were optimised and a detailed material balance has been worked out. Scale-up from laboratory cell to a large scale cell clearly demonstrates the technical feasibility of this process.

Keywords: Electrochemical chlorination, divided cell and monochlorotoluenes

INTRODUCTION

The electrochemical chlorination of toluene to monochlorotoluenes have been carried out by many workers [3-5]. However there are wide variations in the operating conditions in the processes referred. It was therefore, considered necessary to study the optimum conditions such as better cell design, mass transfer, electrode, current density and temperature [7]. In this paper, we present our work on the design of electrochemical cell for electrochemical chlorination of toluene using TSIA electrode (TSIA is titanium substrate insoluble anode) in aqueous hydrochloric acid medium in high yield and low energy consumption [6]. The process conditions have been optimised and detailed material balance studies have been worked out. However, for large scale electrochemical cell it was necessary to devise better electrochemical cell. This electrochemical cell has the narrow and constant inter electrode gap, good agitation, good mass transfer, efficient heat removal. This can be used for the synthesis of various chloro aromatic compounds.

EXPERIMENTAL

Laboratory scale cell

The cell assembly consisted of a glass beaker of 1000 ml capacity acting as an electrochemical cell with a cover made of PVC with suitable provisions to introduce electrodes, stirrer, thermometer and porous diaphragm (Fig. 1). The cell was cooled by using ice and salt in an outer vessel. The anode was TSIA and graphite placed inside the porous diaphragm was used as a cathode. The anolyte aqueous hydrochloric acid containing toluene was agitated by a glass stirrer at 250-300 rpm. The product

monochlorotoluenes having a higher density, settled at the bottom of the cell which was easily separated from the electrolyte. This cell is suitable for gram scale preparation and can be easily scaled up further.

Large laboratory scale cell

The 100 Amp electrolytic cell is made of glass/polypropylene cylindrical cell of about 20 litre capacity with provisions for electrodes, porous diaphragms, stirrer and cooling coil. Good agitation is achieved by a mechanical

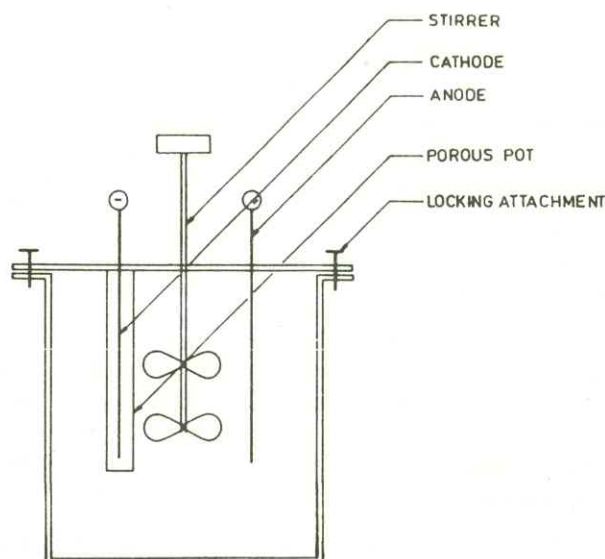


Fig. 1: Electrochemical cell for chlorination

TABLE I: Electrochemical chlorination of toluene in laboratory scale cells

No	Type of cell	Current density (Amp/dm ²)	Temperature K	Yield (%)
1.	divided	2	293-298	81.5
2.	divided	3	293-298	83.0
3.	divided	5	318-323	84.4
4.	divided	5	293-298	86.5
5.	undivided	5	293-298	63.4
6.	undivided	5	293-298	65.0

TABLE II: Electrochemical chlorination of toluene in large scale 100 amp. divided cell/temperature 293-298 K, current density = 5 amp/dm²

No	Cell Voltage V	Amount of product (gms)	Yield (%)	Energy consumption kwhr/kg
1.	4.2	1178	82	2.14
2.	4.0	1185	83	2.03
3.	4.0	1200	84	2.00
4.	4.1	1195	84	2.06

stirrer. About 1.2 kg of monochlorotoluenes could be prepared per batch of operation. The operating cell voltage was 4-6 volts, yield 85% and the power consumption for electrolysis was 2.0-2.14 kwhr/kg

RESULTS AND DISCUSSION

The operating conditions and the results of typical synthetic runs are shown in Table I. Large laboratory scale cell data and results are also shown in Table II. The large scale cell results are substantially consistent with those obtained in the laboratory scale cells. The product monochlorotoluene is a mixture of both orthochlorotoluene and parachlorotoluene and their ratio is about 2:1. Chlorotoluenes both ortho and para are used as key intermediate in the manufacture of pesticides, pharmaceuticals, peroxides, dye and other intermediates [1,2].

Table I shows the effect of temperature and current density in divided cell and undivided cell. The current efficiency and percentage product does not vary very much with increase in temperature and increase in current density.

In case of undivided cell the product yield is lesser than the divided cell. However it will be more advantageous in further scale-up because it avoids the problem of separator in cell design.

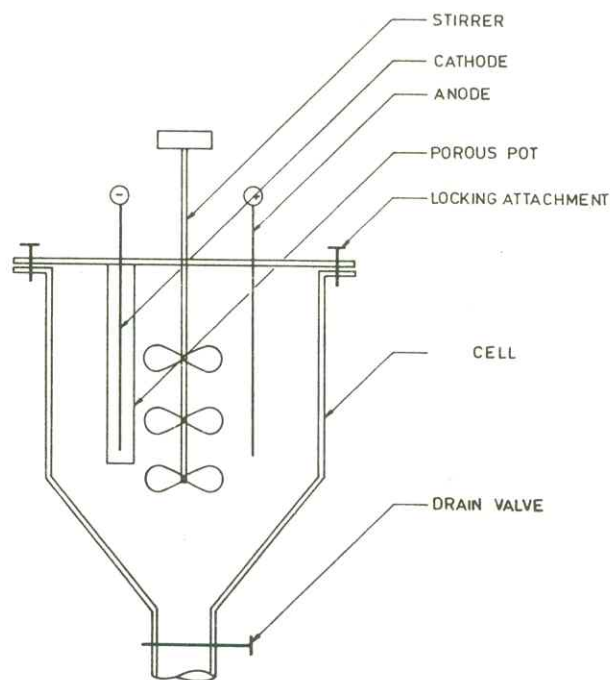


Fig. 2: Electrochemical cell for chlorination

Table II shows the cell voltage, yield of monochlorotoluene and energy consumption of the large laboratory scale cell runs.

CONCLUSIONS

The present study has demonstrated the technical feasibility of the electrochemical cell for electrochemical chlorination of toluene. This electrochemical cell has numerous advantages towards high yields, low energy consumption, pollution free technique and avoid the hazardous chlorine cylinders handling. On the basis of the results of large laboratory scale trial runs, we were able to proceed further in the design of flow cell (Fig. 2) which are useful for the synthesis of important chloro aromatic compounds.

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