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A filter-press electrolytic cell with semi-continuous renewal of sacrifical electrodes

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1. Introduction

The use of sacrificial electrodes in electrochemical syntheses is described frequently in the scientific and patent literature. Several types of cells have been proposed for electrolytic processes in non-aqueous media, in which consumable electrodes are used. A considerable effort has been put into the design of these cells, in order to adapt them to the particular reaction to be processed [1–4]. The peculiarities in the design on the one hand ensure the best performances for that particular process, but, on the other hand, lead to a heavy increase in the investment costs for the electrochemical plant to be installed. In our opinion the availability of multipurpose cells, easily adaptable to processes with or without sacrificial electrodes, should lower the investment costs, making the adoption of this technology more attractive.

Our laboratory has been involved in research on the fundamental and applied aspects of the use of sacrificial metallic anodes in electrocarboxylation processes [5–9]. To scale up some of these processes we have chosen to use a cell of the filterpress type. In fact cells of this type are the most common among those commercially available, and are produced by several firms in different versions. Therefore this type of cell appears to be the most suitable for use in short and diversified production cycles. In order to use these cells in processes in which one of the electrodes is consumed we have designed and set up a device, described here, by which the sacrificial elements can be replaced without the need of disassembling the cell.

2. Experimental

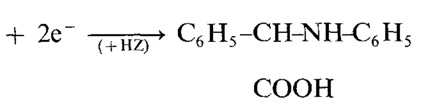
The device is shown in Fig. 1. It consists of a metallic plate ((1) in the case of a central element, and (5) in the case of a side one), and has one or two rubber spacers of suitable shape and thickness. The metallic plates, preferably made of the same metal to be used as sacrificial anode, act as current feeders to the particulate bed, and in the upper part, as containers of the sacrificial elements (see Fig. 1a). The consumable elements (9) are fed through the upper hole (3) (which can be closed by a suitable screw) and descend in the small space (4) and, for central elements, descend on both sides of the plate. On each side of the central plate there is an ethylene–propylene rubber (EPR) spacer (2), approximately 8 mm thick, shaped in such a way that the consumable elements descending from (3) are kept in front of the cathode. A net (10) prevents contact of the consumable elements with the cathodic surface. In our prototype the nets are made of glass fibre covered with PTFE. The whole arrangement of the cell is not altered, the same system of flow distribution (not shown in the figure), and cathodic current feeders being used. In the example shown here, a home made filter-press cell was used, but any commercially available plate and frame cell can be adapted to this device.

The particulate bed consisted of aluminium cylinders 8–10 mm long, with a diameter of 4 mm. A reasonably homogeneous flow distribution was observed in blank experiments (stepwise addition of dyes to the moving medium) in which a polymethylmethacrylate window was used instead of the cathode.

The cell was arranged as shown in Fig. 1b, with two zinc cathodes, a central anodic element, and two anodic end elements. A parallel flow of the circulating solution was imposed, and the electrodes were connected in a monopolar mode. The flow distribution system inside the spacers (6) is not shown in detail. A regular consumption of the exposed surface of the aluminium particles was observed, and, in long term electrolyses, occasional movement of small aluminium particles took place. No short circuits took place in the course of the electrolyses. The apparatus, schematized in Fig. 2, is used in our laboratory for the synthesis of some carboxylic acids from different starting materials.

3. Results and discussion

The cell was tested for the scale-up of the electrocarboxylation of benzalaniline to the corresponding α-amino acid according to the stoichiometry of Reaction 1.



$$C_6H_5\text{-}CH{=}N\text{-}C_6H_5 + CO_2 + 2e^- \xrightarrow[(+HZ)]{} \underset{\displaystyle COOH}{C_6H_5\text{-}CH\text{-}NH\text{-}C_6H_5} \qquad (1)$$

By-products of the synthesis are a dimerization derivative and the product of hydrogenation of the

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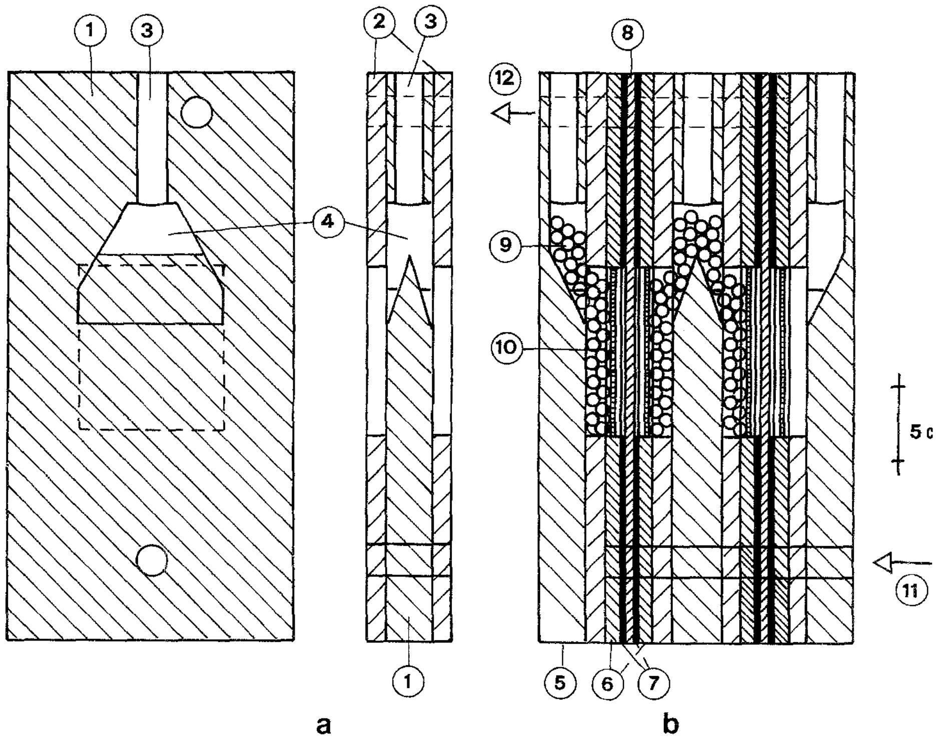

Fig. 1. a) Cross-sections of a central element; b) Cross section of an assembled cell. 1 – Central aluminium plate. 2 – EPR spaces. 3 – Threaded upper hole. 4 – Space for the particulate bed. 5 – Side aluminium plate. 6 – Net holder and flow distributor. 7 – EPR gaskets. 8 – Cathode. 9 – Particulate bed. 10 – Net. 11 – Fluid inlet. 12 – Fluid outlet.

imine. As previously reported [10, 11], the use of aluminium sacrificial anodes in diaphragmless cells leads to substantial improvements in the yields with respect to the conventional electrosynthetic methods.

The operating data and the results of a typical synthetic run are shown in Table 1.

These results are substantially consistent with those obtained in small scale experiments, but with the advantage of the use of a cell with an average interelectrodic gap remaining constant for the whole run, without the need of disassembling the cell when the anodic material is consumed. In fact for several consecutive runs the consumed anodic material was replaced by introduction, through the upper holes, of the corresponding amount of aluminium, and a reproducible electrical behaviour of the cell was always observed.

Tests on the behaviour of various commercial filter-press cells to which this device has been successfully adapted are now being performed and will be the subject of further communications.

Acknowledgements

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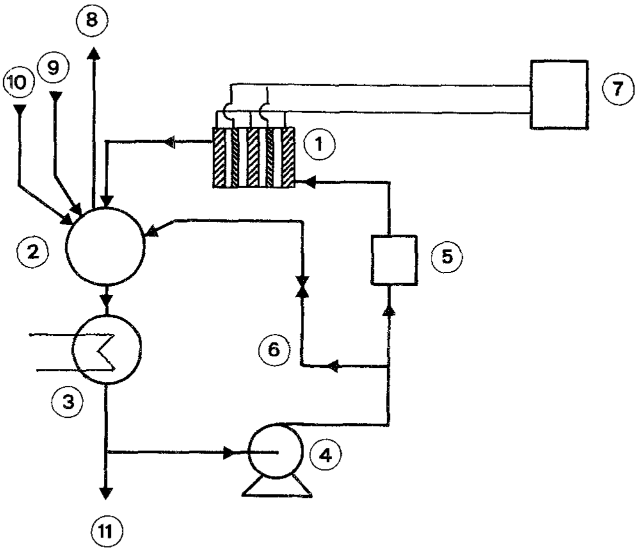

Fig. 2. Simplified flow-sheet of the electrolysis apparatus. 1 – Electrolysis cell. 2 – Solution tank and carbon dioxide saturator. 3 – Heat exchanger. 4 – Pump. 5 – Flow meter. 6 – By-pass. 7 – Power supply. 8 – Purge. 9 – Carbon dioxide inlet. 10 – Electrolytic solution inlet. 11 – Electrolytic solution outlet.

Table 1. Electrocarboxylation of N-phenyl-benzalimine

N-phenyl-benzalimine: 200g;
Solvent: *N,N*-dimethylformamide;
Supporting electrolyte: tetrabutylammonium bromide (3 wt %);
Overall volume of circulating solution: 2 l;

Cathode: Zn foil (99.5 grade);
Cathodic surface: 400 cm^2;
Anode: Al cylinders (99.8 grade);
Anodic initial working surface area: ~ 500 cm^2;

Overall current: 8–9 A;
Overall applied voltage: 7–9 V;
Flow rate: 15–22 l min^{-1};
Temperature: 20° C;

Converted imine: 92%
Product yield: 85%;
Current yield: 80%;
Anodic current efficiency: 108%.

The cell used for this synthesis was arranged as in Fig. 2. The procedure of isolation of the product was already described (see Reference 11).

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