GOVERNMENT OF INDIA : THE PATENT OFFICE, 214, LOWER CIRCULAR ROAD, CALCUTTA-17.
Specification No. 102485. Application No. 102485, dated 12th November 1965. Complete Specification left on 6th August 1966. (Application accepted 11th July 1967.)
Index at acceptance—32F3b[IX(1)], 70C5[LVIII(5)].

IMPROVEMENTS IN OR RELATING TO THE ELECTROCHEMICAL PRODUCTION OF SUCCINIC ACID FROM EITHER MALEIC ACID OR FUMARIC ACID.

PROVISIONAL SPECIFICATION

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, Rafi Marg, New Delhi-1, India, an Indian registered body incorporated under the Registration of Societies Act (Act XXI of 1860).

The following specification describes the nature of this invention.

This is an invention by HANDADY VENKATAKRISHNA UDUPA, Scientist, MYSORE SEETHAIYER VENKATACHALAPATHY, Scientist and RAMANUJA IYENGAR KANAKAM, Senior Laboratory Assistant, all of the Central Electrochemical Research Institute, Karaikudi, all Indian citizens.

This invention relates to improvements in or relating to the electrochemical production of succinic acid from either maleic acid or fumaric acid.

Hitherto it has been proposed to reduce electrolytically either fumaric acid or maleic acid using a solution of either maleic or fumaric in dilute sulphuric acid at either mercury or amalgamated lead cathode (stationary) by employing separate cathode and anode chambers which are separated by means of a diaphragm.

This is open to the objection in that the process reported in literature suffers from the following drawbacks: (1) the choice of a proper anode chamber complicates the process and (2) only low current density can be employed at a stationary cathode.

The object of this invention is to obviate these disadvantages by the modifications and improvements now effected by us. According to the present invention, separate anode chamber and anolyte are avoided by using an anode closely wrapped with asbestos fibre, yarn or cloth and using a rotating lead cathode. The present process is superior to the processes hitherto described in literature since it not only overcomes use of the troublesome and costly anode chamber suggested earlier but uses instead of an anode closely fitted by modern diaphragm materials like asbestos and takes into account the drawback of low current density and thereby renders the process more easy to operate without any economic drawbacks. Additional advantage in the process now established by us is that by rotating the cathode, high current density and high concentration of either maleic or fumaric acid could be employed without adversely affecting the energy economics of the process.

To these ends, the invention broadly consists in reducing cathodically either maleic or fumaric acid to give succinic acid in an electrolytic cell, fitted with a rotating lead cathode and lead lined copper anodes, closely covered with asbestos thread. The concentration of maleic acid or fumaric acid was kept between 10 and 30 per cent but a solution of 22 per cent could give the maximum current efficiency and yield. A current density of 2·5-5 amp/dm^2 in the case of stationary cathode and 10-30 amp/dm^2 with a rotating cathode could be employed. The temperature of electrolysis could vary from 50-80°C but a temperature of 70-72°C was employed for most of the experiments. A current efficiency of 98% and an yield of 95% was obtained.

EXAMPLE 1

Electrolytic reduction of maleic acid using a stationary lead cathode:

80 g of maleic acid was dissolved in 350 ml of 5% sulphuric acid. Lead was employed as cathode. Copper rod lined with lead and covered closely with asbestos thread was used as anodes. During the electrolysis the temperature of the electrolyte was kept between 70 and 72°C by means of a water bath. A current density of 2·5 amp/dm^2 was employed. After passing 37 amp-hrs, 73 g of succinic acid was obtained with a current efficiency of 100% and an yield of 91·25%.

EXAMPLE 2

Electrolytic reduction of maleic acid using a rotating lead cathode:

21 g of maleic acid was taken in 230 ml of 5% sulphuric acid. A rotating cylinder of lead was employed as cathode. The r.p.m. was kept between 1000 and 1200. During the electrolysis, the temperature of the electrolyte was kept between 70 and 72°C. A current density of 20 amp/dm^2 was employed and after passing 9·6 amp-hr, 20 g of succinic acid was obtained. No unreduced maleic acid could be detected polarographically. A current efficiency of 100% and an yield of 95% was obtained. The anode employed was the same as given in Example 1.

EXAMPLE 3

Electrolytic reduction of fumaric acid using a rotating lead cathode:

21 g of fumaric acid was dissolved in 200 ml of 5% sulphuric acid. A rotating cylinder of lead was employed as cathode. The current density employed was 10 amp/dm^2 and all the other details were the same as given in Example 2. When the reaction was over, the electrolyte was filtered while hot and evaporated to the point of its crystallisation, cooled and the crystalline solid separated was filtered and air dried. 20 g of succinic acid was obtained with an yield of 95%.

The following are among the main advantages of the invention:

(1) A process for the electrochemical preparation of succinic acid either from maleic or fumaric acid using a lead cathode either stationary or rotating and having an anode on which asbestos has been closely wrapped as diaphragm.

(2) Copper rods lined with lead and closely covered with asbestos material are used as anodes.

(3) A separate anode chamber with dilute sulphuric acid is avoided.

(4) A current density of 2·5-5 amp/dm^2 but preferably 3 amp/dm^2 in the case of stationary cathode and 10-30 amp/dm^2 in the case of rotating cathode are employed to obtain succinic acid and in good yield (95-96%).

(5) The percentage of aqueous solution either maleic acid or fumaric acid could be kept between 10 and 30 but preferably 22 per cent.

(6) The concentration of sulphuric acid in the electrolyte could be varied between 5 and 10% but preferably 5%.

(7) The temperature range could be 50-80°C but preferably at 70-72°C.

(8) The cathode is stationary or rotating and while stationary an auxiliary stirrer is employed.

R. BHASKAR PAI

Patents Officer,

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH.

Dated this 8th day of November 1965.

Price: TWO RUPEES.

COMPLETE SPECIFICATION

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAFI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860).

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This is an invention by HANDADY VENKATAKRISHNA UDUPA, Scientist, MYSORE SESHAIYER VENKATACHALAPATHY, Scientist and RAMANUJA IYENGAR KANAKAM, Senior Laboratory Assistant, all of the Central Electrochemical Research Institute, Karaikudi, all Indian citizens.

This invention relates to improvements in or relating to the electrochemical production of succinic acid from either maleic acid or fumaric acid.

Hitherto it has been proposed to reduce electrolytically either fumaric acid or maleic acid using a solution of either maleic or fumaric in dilute sulphuric acid at either mercury or amalgamated lead cathode (stationary) by employing separate cathode and anode chambers which are separated by means of a diaphragm.

This is open to the objection in that the process reported in literature suffers from the following drawbacks: (1) the choice of a proper anode chamber complicates the process and (2) only low current density can be employed at a stationary cathode.

The object of this invention is to obviate these disadvantages by the modifications and improvements now effected by us. We have found that separate anode chamber and anolyte can be avoided by using an anode closely wrapped with asbestos fibre, yarn or cloth and using a rotating lead cathode. The present process is superior to the processes hitherto described in literature since it not only overcomes use of the troublesome and costly anode chamber suggested earlier but uses instead an anode closely fitted by modern diaphragm materials like asbestos and takes into account the drawback of low current density and thereby renders the process more easy to operate without any economic drawbacks. Additional advantage in the process now established by us is that by rotating the cathode, high current density and high concentration of either maleic or fumaric acid could be employed without adversely affecting the energy economics of the process.

According to the present invention, the process for the electrochemical preparation of succinic acid consists in the reduction of maleic or fumaric acid in sulphuric acid electrolyte using a lead or lead plated cathode, either stationary or rotating, and a lead anode on which asbestos has been closely wrapped as diaphragm.

Thus, copper rods covered with lead and closely wound with asbestos rope, fibre or cloth material are used as anodes.

A cathodic current density of 2·5–5 amp/dm² but preferably 3 amp/dm² is used in the case of stationary cathode and 10-30 amp/dm² but preferably 20 amp/dm² is used when the cathode is rotating in order to obtain succinic acid with an yield of 95-96%.

The strength of aqueous solution of maleic acid or fumaric acid is kept between 10% and 30% but preferably at 22%.

The concentration of sulphuric acid in the electrolyte is between 5% and 10%, preferably 5%.

The reduction is carried out at a temperature range of 50°-80°C, preferably at 70-72°C.

An auxiliary stirrer is employed when a stationary cathode is used.

Either maleic or fumaric acid is reduced cathodically to give succinic acid in an electrolytic cell, fitted with a rotating lead cathode and lead lined copper anodes, closely covered with asbestos thread. The concentration of maleic acid or fumaric acid was kept between 10 and 30 per cent but a solution of 22 per cent could give the maximum current efficiency and yield. A current density of 2·5-5 amp/dm² in the case of stationary cathode and 10-30 amp/dm² with a rotating cathode could be employed. The temperature of electrolysis could vary from 50-80°C but a temperature of 70-72°C was employed for most of the experiments. A current efficiency of 98% and an yield of 95% was obtained.

EXAMPLE 1

Electrolytic reduction of maleic acid using a stationary lead cathode:

80 g of maleic acid was dissolved in 350 ml of 5% sulphuric acid. Lead was employed as cathode. Copper rod lined with lead and covered closely with asbestos thread was used as anodes. During the electrolysis the temperature of the electrolyte was kept between 70 and 72°C by means of a water bath. A current density of 2·5 amp/dm² was employed. After passing 37 amp-hrs, 73 g of succinic acid was obtained with a current efficiency of 100% and an yield of 91·25%.

EXAMPLE 2

Electrolytic reduction of maleic acid using a rotating lead cathode:

21 g of maleic acid was taken in 230 ml of 5% sulphuric acid. A rotating cylinder of lead was employed as cathode. The r.p.m. was kept between 1000 and 1200. During the electrolysis, the temperature of the electrolyte was kept between 70 and 72°C. A current density of 20 amp/dm² was employed and after passing 9·6 amp-hr, 20 g of succinic acid was obtained. No unreduced maleic acid could be detected polarographically. A current efficiency of 100% and an yield of 95% was obtained. The anode employed was the same as given in Example 1.

EXAMPLE 3

Electrolytic reduction of fumaric acid using a rotating lead cathode:

21 g of fumaric acid was dissolved in 200 ml of 5% sulphuric acid. A rotating cylinder of lead was employed as cathode. The current density employed was 10 amp/dm² and all the other details were the same as given in Example 2. When the reaction was over, the electrolyte was filtered while hot and evaporated to the point of its crystallisation, cooled and the crystalline solid separated was filtered and air dried. 20 g of succinic acid was obtained with an yield of 95%.

We claim:

1. A process for the electrochemical preparation of succinic acid which consists in the reduction of maleic or fumaric acid in sulphuric acid electrolyte using a lead or lead plated cathode, either stationary or rotating, and a lead anode on which asbestos has been closely wrapped as diaphragm.

2. A process as claimed in Claim 1 wherein copper rods covered with lead and closely wound with asbestos rope, fibre or cloth material are used as anodes.

3. A process as claimed in Claim 1 wherein a cathodic current density of 2·5–5 amp/dm² but preferably 3 amp/dm² is used in the case of stationary cathode and 10-30 amp/dm² but preferably 20 amp/dm² is used when the cathode is rotating in order to obtain succinic acid with an yield of 95-96%.

4. A process as claimed in Claim 1 wherein the strength of aqueous solution of maleic acid or fumaric acid is kept between 10% and 30% but preferably at 22%.

5. A process as claimed in Claim 1 wherein the concentration of sulphuric acid in the electrolyte is between 5% and 10%, preferably 5%.

6. A process as claimed in Claim 1 wherein the reduction is carried out at a temperature range of 50°-80°C, preferably at 70-72°C.

7. A process as claimed in Claim 1 wherein an auxiliary stirrer is employed when a stationary cathode is used.

8. Succinic acid whenever obtained by the electrochemical reduction of fumaric or maleic acid according to the process substantially as hereinbefore described.

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Dated this 3rd day of August 1966.

MGIP Sant.—S3—46 Patent/68—9-12-69—100.