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Group—B

IMPROVEMENTS IN OR RELATING TO THE ELECTROLYTIC PREPARATION OF LITHIUM HYDROXIDE.

PROVISIONAL SPECIFICATION.

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, RAFI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED SOCIETY 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, Director, RAMASWAMY THANGAPPAN NADAR, Scientist, PERUMAL SUBBIAH, Junior Scientific Assistant, all of Central Electrochemical Research Institute, Karaikudi, India, all Indians.

This invention relates to improvements in or relating to the electrolytic preparation of lithium hydroxide suitable for use as absorbent for carbon dioxide, in alkaline storage batteries and in the manufacture of lithium stearate and other lithium soaps.

Hitherto it has been proposed to prepare lithium hydroxide by chemical methods or electrolysing an aqueous solution of lithium chloride or sulphate in mercury cell or diaphragm cell using platinum or graphite as the anode.

This is open to the objection that the concentration of lithium hydroxide attainable in solution is only about 20 g|l and the product is not free from impurities like alumina, lime, iron etc., in the chemical methods. One of the chemical methods uses lithium carbonate as the starting material which itself is an end product obtained in a lengthy extraction process. In the mercury process, the cost involved in the inventory of mercury is high and the cost of plantinum loss due to wear and tear is quite significant. The current efficiency is low while using graphite as the anode and the disintegration of graphite is severe.

The object of this invention is to obviate these disadvantages by electrolysing an aqueous solution of lithium chloride in a vertical diaphragm cell with unsubmerged nickel plated steel cathode and lead dioxide anode.

To these ends, the invention broadly consists in the electrolysis of an aqueous solution of lithium chloride (100 to 500 g|l) in a cylindrical cell with conical bottom with a cylindrical lead dioxide anode at the centre separated from the nickel plated perforated steel cathode by asbestos diaphragm fixed along the surface facing the anode. The above said process of electrolysis is carried out by continuously feeding the electrolyte to the anode compartment and allowing to percolate into the cathode chamber through the diaphragm. The catholyte liquor, a mixture of lithium hydroxide and unconverted lithium chloride, drip off from the surface of the cathode as soon as it is formed. The temperature of electrolysis is maintained at desired values between 40 and 95°C. The current density employed is in the range of 6-15 amp|dm². The concentration of lithium hydroxide in the catholyte liquor is maintained at 25-100 g|l.

The following typical Examples are given to illustrate the invention :

Example I

Anode	Cylindrical graphite substrate lead dioxide
Cathode	Nickel plated perforated steel
Diaphragm . . .	Asbestos
Current passed . .	5 amps
Anode current density .	10·42 amp/dm²
Concentration of lithium chloride in the feed.	499.8 g/l
Concentration of lithium hydroxide in the catholyte.	34.6g/l
Temperature of electrolysis	57±1°C
Average cell voltage .	3·4 volts
Duration of electrolysis .	4 hours
Volume of catholyte .	490 ml
Current efficiency . .	94·6%
Energy consumption per kg of lithium hydroxide formed.	4·01 Kwh

Example II

Anode	Cylindrical graphite substrate lead dioxide
Cathode	Nickel plated perforated steel
Diaphragm . . .	Asbestos
Current passed . .	6 amps
Anode current density .	12·5 amp/dm²
Concentration of lithium chloride in the feed.	325 g/l
Concentration of lithium hydroxide in the catholyte.	78 g/l
Temperature of electrolysis	50±1°C
Average cell voltage .	3·64 volts
Duration of electrolysis .	4 hours
Volume of catholyte .	257 ml
Current efficiency . .	93·1%
Energy consumption per kg of lithium hydroxide formed.	4·36 Kwh

The following are among the main advantages of the invention :

1. By employing the above said combination of lead dioxide anode and unsubmerged cathode, very high current efficiency of 85 to 95% is obtained at anode current densities of 6 to 15 amp|dm².
2. The use of insoluble lead dioxide anode eliminates frequent replacement of anodes and hence reduces down times and operating cost.
3. This process could be adopted for continuous operation.
4. As the starting material is commercially pure lithium chloride, the product obtained is free from impurities like alumina, lime and iron, chloride being removed by fractional crystallisation.

R. BHASKAR PAI

Patents Officer,

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH,

Dated this 26th day of June 1971.

Price: TWO RUPEES.

COMPLETE SPECIFICATION

COUNCIL OF SCIENTIFIC & 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, Director, RAMASWAMY THANGAPPAN NADAR, Scientist and PERUMAL SUBBIAH, Junior Scientific Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, India, all Indian citizens.

This invention relates to improvements in or relating to the electrolytic preparation of lithium hydroxide suitable for use as absorbent for carbon dioxide, in alkaline storage batteries and for the manufacture of lithium stearate and other lithium soaps.

Hitherto it has been proposed to prepare lithium hydroxide by chemical methods or electrolysing an aqueous solution of lithium chloride or sulphate in mercury cell or diaphragm cell using platinum or graphite as the anode.

This is open to the objection that the concentration of lithium hydroxide attainable in solution is only about 20 g|l and the product is not free from impurities like alumina, lime, iron, in the chemical methods. One of the chemical methods uses lithium carbonate as the starting material which itself is an end product obtained in a lengthy extraction process. In the mercury process, the cost involved in the inventory of mercury is high and the cost of platinum loss due to wear and tear is quite significant. The current efficiency is low while using graphite as the anode and the disintegration of graphite is severe.

The object of this invention is to obviate these disadvantages by electrolysing an aqueous solution of lithium chloride in a vertical diaphragm cell with unsubmerged nickel plated steel cathode and the lead dioxide anode.

The new principle underlying the invention is the employment of lead dioxide anode in a diaphragm type cell with unsubmerged cathode for the preparation of pure lithium hydroxide by elctrolysing an aqueous solution of commercially pure lithium chloride at current densities between 6 and 15 amp|dm².

According to the present invention, there is provided a process for the production of lithium hydroxide which consists in the electrolysis of an aqueous solution of lithium chloride (200 to 500 g|l) in a cylindrical cell with conical bottom characterised in that the cell in which the electrolysis is carried out is provided with cylindrical lead dioxide anode at the centre separated from a nickel plated perforated steel cathode by asbestos diaphragm fixed along the surface facing the anode.

Lithium hydroxide upto a concentration of 100 g|l in the catholyte was obtained in the process at current densities upto 15 amp|dm². The current efficiency varied between 85 and 95% and the energy consumption varied between 3.6 and 5.5 kwh|kg of lithium hydroxide formed, depending upon the operating conditions maintained. The use of insoluble lead dioxide anode eliminated frequent replacement of anodes and the weight loss of the anode for the passage of a current of 796 amp-hr. was only 0.71 g. Since the starting material was commercially pure lithium chloride, the product obtained was free from impurities like alumina, lime and iron, chloride being removed by fractional crystallisation.

To these ends, the invention broadly consists in the electrolysis of an aqueous solution of lithium chloride (200 to 500 g|l) in a cylindrical cell with conical bottom with a cylindrical lead dioxide anode at the centre separated from the unsubmerged nickel plated perforated steel cahode by asbestos diaphragm fixed along the surface facing the anode. The above said process of electrolysis is carried out by continuously feeding the electrolyte to the anode compartment and allowing it to percolate into the cathode chamber through the diaphragm. The catholyte liquor, a mixture of lithium hydroxide and unconverted lithium chloride, drip off from the surface of the cathode as soon as it is formed. The temperature of electrolysis is maintained at desired values between 40 and 90°C. The current density employed is in the range of 6-15 amp|dm². The concentration of lithium hydroxide in the catholyte liquor is maintained at 25-100 g|l.

The flow sheet of the process is given in Figure 1 of the accompanying drawings. Lithium chloride electrolyte at the required concentration is stored in the reservoir (1) from where it flows into the anode compartment of the electrolytic cell (4) through the constant level tank (3). The temperature of the electrolyte is maintained at the desired value by the immersion silica heater (2). Current to the cell is supplied by a selenium rectifier (R) and ammeter (A) and voltmeter (V) are connected in the circuit to measure the cell current and cell voltage respectively. The anolyte percolates through the diaphragm into the cathode surface where lithium hydroxide and hydrogen are formed. Hydrogen escapes through the hydrogen outlet provided at the top of the cell and the catholyte containing lithium hydroxide and unconverted lithium chloride, in the form of film over the entire surface of the cathode falls as droplets into the receiver (5). Chlorine evolved at the anode is removed by applying a low suction of 0.5 cm of water and absorbing it in lime slurry. The cell effluent from the receiver flows into the storage vessel (6) from where it is tapped periodically for processing. The effluent is concentrated by evaporation in an evaporating vessel (7). Subsequently it is cooled and the lithium hydroxide monohydrate crystals formed are separated by centrifuging (9). The filtrate from the centrifuge still contains about 50% of the lithium hydroxide formed in the cell. This solution is saturated with carbon dioxide in a carbonating vessel (10) and the lithium carbonate formed is separated in the centrifuge (11). The filtrate from the centrifuge is a weak solution of lithium chloride and is resaturated with additional quantity of lithium chloride and water in the saturator (12) from where it is pumped (13) back to the electrolyte reservoir.

The electrolytic cell (4) with cylindrical body and conical bottom is provided at the centre with the lead dioxide anode (14). The cathode (15) and diaphragm (16) rolled in the form of a cylinder with closed bottom surrounds the anode. Provisions are given in the cell for brine inlet (17), chlorine outlet (18), hydrogen outlet (19) and catholyte outlet (20).

The following typical Examples are given to illustrate the invention :

Example 1

Anode	Cylindrical graphite substrate lead dioxide anode
Cathode	Nickel plated perforated stee
Diaphragm	Asbestos
Current passed	5 amps
Anode current density	10·42 amp/dm²
Concentration of lithium chloride in the feed.	499·8 g/l
Concentration of lithium hydroxide in the catholyte.	34·6 g/l
Temperature of electrolysis	57±1°C
Average cell voltage	3·4 volts
Duration of electrolysis	4 hours
Volume of catholyte	490 ml
Current efficiency	94·6%
Energy consumption per kg of lithium hydroxide formed.	4·01 Kwh

Example II

Anode	Cylindrical graphite substrate lead dioxide
Cathode	Nickel plated perforated steel
Diaphragm . . .	Asbestos
Current passed . .	6 amps
Anode current density .	12·5 amp/dm²
Conc. of lithium chloride in the feed.	325 g/l
onc. of lithium hydroxide in the catholyte.	78 g/l
Temperature of electrolysis	50±1°C
Average cell voltage .	3·64 volts
Duration of electrolysis .	4 hours
Volume of catholyte .	257 ml
Current efficiency . .	93·1%
Energy consumption per kg of lithium hydroxide formed.	4.36 Kwh

The advantages of the invention are :

(1) By employing the above said combination of lead dioxide anode and unsubmerged cathode, very high current efficiency of 85 to 95% is obtained at anode current densities of 6 to 15 amp|dm².

(2) The use of insoluble lead dioxide anode eliminates frequent replacement of anodes and hence reduces down times and operating cost.

(3) This process could be adopted for continuous operation.

(4) The product obtained is free from impurities like alumina, lime and iron, chloride being removed by fractional crystallisation.

Summarising, the present invention affords a convenient process for the preparation of lithium hydroxide by electrolysing an aqueous solution of lithium chloride in a vertical diaphragm cell with unsubmerged nickel plated steel cathode and lead dioxide anode.

We claim :

1. A process for the production of lithium hydroxide which consists in the electrolysis of an aqueous solution of lithium chloride (200 to 500 g|l) in a cylindrical cell with conical bottom characterised in that the cell in which the electrolysis is carried out is provided with cylindrical lead dioxide anode at the centre separated from a nickel plated perforated steel cathode by asbestos diaphragm fixed along the surface facing the anode.

2. A process as claimed in Claim 1 wherein the current density employed is in the range of 6-15 amp|dm² and temperature 40-90°C.

3. A process as claimed in the preceding claims wherein the cathode is kept unsubmerged.

4. A process for the preparation of lithium hydroxide, substantially as described hereinbefore in the examples and in Figure 1.

5. A process for the preparation of lithium hydroxide suitable for use as absorbent for carbon dioxide, in alkaline storage batteries and in the manufacture of lithium stearate and other lithium soaps substantially as hereinbefore described.

R. BHASKAR PAI

Patents Officer,

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH.

Dated this 23rd day of February 1972.

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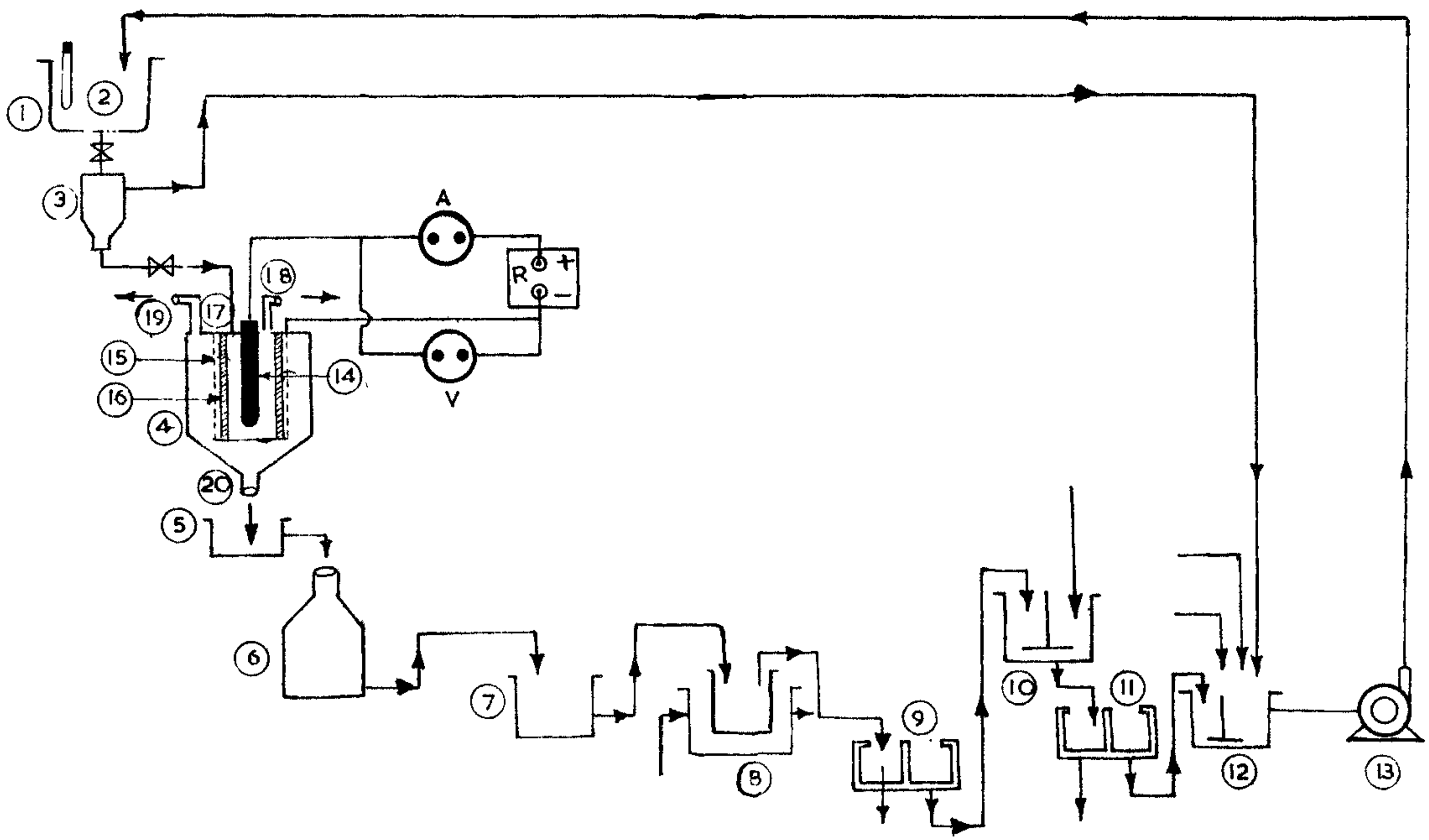

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