(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 338 719**
A1

(12) EUROPEAN PATENT APPLICATION

(21) Application number: **89303609.5**

(51) Int. Cl.[4]: **C22B 26/10**

(22) Date of filing: **12.04.89**

(30) Priority: **20.04.88 GB 8809355**

(43) Date of publication of application:
25.10.89 Bulletin 89/43

(84) Designated Contracting States:
DE FR GB

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(54) A process for the separation of sodium and calcium from sodium sludge.

(57) A process for the separation of sodium from a sludge containing sodium, calcium and oxides thereof which comprises the step of subjecting the sludge to a centrifugal force to separate the sodium from the calcium.

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A PROCESS FOR THE SEPARATION OF SODIUM AND CALCIUM FROM SODIUM SLUDGE

The present invention relates to a process for the separation of sodium and calcium from sodium sludge and, in particular, to the separation of sodium and calcium from the sludge produced as a waste product in the production of sodium by the electrolysis of a fused mixture of sodium chloride and calcium chloride.

The sludge produced in the above process generally has a composition comprising 70% Na, 20% Ca and 10% mixed oxides but mainly Na_2O. Currently, only sodium is separated and recycled and the calcium is not recovered.

We have now developed a process for the separation of sodium from a sludge containing sodium and calcium. The process also enables the calcium remaining after separation of the sodium from the sludge to be recovered.

Accordingly, the present invention provides a process for the separation of sodium from a sludge containing sodium, calcium and oxides thereof which comprises the step of subjecting the sludge to a centrifugal force to separate the sodium from the calcium.

The sludge is preferably restrained in a gauze cage made from a material which is not attacked by the sludge, for example stainless steel. The gauze may have a mesh size of from 25 to about 200 micrometres, preferably 25 to 125 micrometres. The gauze cage containing the sludge is preferably immersed in an oil bath maintained at an elevated temperature and preferably at a temperature in the range of from 110° to 200° C. The liquid used for heating can be any liquid that is non reactive with the sludge over the said temperature range, for example, mineral oils, kerosene (deodorised or otherwise) and liquid paraffin (light and heavy fractions). The gauze cage assembly can be preheated to the working temperature prior to loading with the sludge.

The gauze cage may be rotated at a rate of up to 30,000 r.p.m., preferably 600 to 3,000 r.p.m., more preferably 2,000 to 3,000 r.p.m., in order to cause the sodium to separate from the calcium. The centrifugal force applied to the sludge causes the sodium to migrate to the edge of the gauze cage and to be flung through the gauze as a plurality of discrete particles which collect at the bottom of the oil bath. At rotational speeds of above about 600 r.p.m., the separation of the sodium from the sludge is substantially complete. The centrifugal separation may be carried out under an atmosphere of an inert gas, for example, nitrogen.

The sludge treated according to the process of the invention is preferably a sludge containing 70% sodium, 20% calcium and up to 10% of sodium oxide.

In order to break up the sludge during the centrifugal spinning, particles of an inert material may be added to the sludge. The particles may be of a regular or irregular shape and may be, for example, glass beads or irregularly shaped glass particles, ball bearings, polymer beads or ceramic particles.

The residue remaining after the separation of the sodium from the sludge may, if desired, be treated by one of the following methods to separate the calcium therefrom:-

i) the residue may be mixed with a mixture of an alcohol and water, preferably methanol, ethanol or propanol and water in a ratio of 65 to 85 arts of methanol, ethanol or propanol to 35 to 15 parts of water. The sodium oxide dissolves in the water and the calcium remaining is then filtered and dried.

ii) the calcium may be separated from the residue by mixing particles of the residue with a liquid having a density in the range of from 1.6 to 2.0 g/cm^3. A liquid having such a density is between the density of calcium and sodium oxide and, accordingly, the particles of calcium will float to the surface of the liquid, whilst the particles of sodium oxide sink to the bottom of the liquid. Certain halogenated hydrocarbons, such as 1,3-dibromopropane which has a density of 1.9 g/cm^3, are suitable for use. Another non-reacting liquid of density less than 1.6 g/cm^3 may also be employed above the calcium layer in order to protect the calcuim from exposure to the atmosphere. A suitable liquid for this purpose is petroleum spirit fraction at 120 to 160° C, which has a density of 0.75 g/cm^3. Preferably the lower layer of liquid of density greater than 1.6 will generally comprise from 30 to 90% by volume of the total volume of the lower and upper layers.

The present invention will be further described with reference to the following Example. All percentages are by weight unless otherwise stated.

EXAMPLE 1

A sludge was prepared for experimental purposes having a nominal composition of 70%Na, 20%Ca and 10%Na_2O. The constituents of the sludge were heated to about 200°C in order to form a substantially homogeneous product.

Separation of the sludge was carried out by placing the sludge in a stainless steel gauze cage having a mesh size of 125 micrometres. The cage containing the sludge was placed in an oil bath maintained at a temperature of 140°C and rotated at a speed of 1,500 r.p.m. The sodium was found to separate readily from the sludge. The calcium content of the sodium recovered from the sludge was found on an average of three analyses to be 0.044%.

A mixture of Ca:Na_2O of 66:33 was used to check the solubility of the sodium oxide in a water:industrial methylated spirit mixture. 3 grams of the residue was dissolved in 200 ml of the water/alcohol mixture in all cases. The following results were obtained:-

Water:alcohol	Time for Na_2O to dissolve	% Ca recovery
15:85	12 minutes	99
20:80	13 minutes	95
30:70	2 min 35 sec	95
35:65	1 min 18 sec	99

A sample of the calcium/sodium oxide residue was separated using a density separation method with 1,3-dibromopropane as the liquid medium. 1,3-Dibromopropane has a density of 1.9 g/cm^3 which is between the density of calcium (1.55 g/cm^3) and that of sodium oxide (>2.0 g/cm^3). The calcium floated to the top of the liquid whilst the sodium oxide remained at the bottom of the liquid.

EXAMPLE 2

A sludge produced as a by-product of the Downs Cell electrolytic sodium production process was used in carrying out trials of separation. The composition of this sludge was nominally 70% Na, 20% Ca and 10% oxides, mainly Na_2O.

The sludge was placed into a stainless steel gauze cage with a mesh size of 125 micrometres and spun in hot mineral oil at 140°C. Thereafter, centrifugal separation at 3000 rpm was used for up to 3 minutes. The sodium separated from the sludge as discrete droplets through the mesh whilst the calcium and the oxide remained contained within.

The residual calcium and oxides were washed in ethanol to remove all traces of sodium and thereafter placed into a suitable container with 1,3 dibromopropane. Petroleum spirit fraction 120 to 160° was poured on top of the 1,3 dibromopropane to protect the calcium metal from oxidation.

EXAMPLE 3

The procedure of Example 2 was repeated, except that the mineral oil was replaced by a liquid paraffin heavy fraction having a density of 0.86 to 0.89 g/cm^3 maintained at 150°C.

Claims

1. A process for the separation of sodium from a sludge containing sodium, calcium and oxides thereof which comprises the step of subjecting the sludge to a centrifugal force to separate the sodium from the calcium.

2. A process as claimed in claim 1 wherein the sludge is restrained in a gauze cage.

3. A process as claimed in claim 2 wherein the gauze has a mesh size in the range of from 25 to 125 micrometres.

4. A process as claimed in claim 2 or claim 3 wherein the gauze cage containing the sludge is immersed in an oil bath.

5. A process as claimed in claim 4 wherein the oil bath is maintained at a temperature of 110° to 200° C.

6. A process as claimed in any one of claim 2 to 5 wherein the gauze cage containing the sludge is rotated at a rate of up to 30,000 r.p.m, preferably at a rate of from 2,000 to 3,000 r.p.m.

7. A process as claimed in any one of the preceding claims wherein the sludge comprises 70% sodium, 20% calcium and up to 10% of sodium oxide.

8. A process as claimed in any one of the preceding claims wherein regular or irregular particles are added to the sludge prior to the centrifugal spinning.

9. A process as claimed in any one of the preceding claims wherein after separation of the sodium from the sludge the residue is subjected to treatment by mixing particles thereof with a water:alcohol mixture in order to dissolve sodium oxide therefrom.

10. A process as claimed in claim 9 wherein the alcohol is methanol, ethanol or propanol.

11. A process as claimed in claim 9 or claim 10 wherein the ratio of the alcohol to water in the mixture is in the range of from 85:15 to 65:35.

12. A process as claimed in any one of claims 1 to 8 wherein after separation of the sodium from the sludge the residue is subjected to treatment by mixing particles thereof with a liquid having a density in the range of from 1.6 to 2.0 g/cm^3.

13. A process as claimed in claim 12 wherein the liquid is 1,3-dibromopropane.

14. A process as claimed in claim 12 wherein a liquid having a density of less than 1.6 g/cm^3 is provided as a layer above the liquid having a density of 1.6 to 2.0 g/cm^3.

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EUROPEAN SEARCH REPORT

Application Number

EP 89 30 3609

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
Y	US-A-2 689 791 (BOAG) * column 3; column 5, line 29 *	1	C 22 B 26/10
Y	US-A-3 428 447 (ADDIS) * claim 11 *	1	
A	US-A-2 759 896 (HAWKES et al.) * column 3, line 3 *	9	
A	US-A-1 943 307 (GILBERT) * page 2, line 50; claim 1 *	1	
A	FRANZ PAWLEK: METALLHUETTENKUNDE Walter de Gruyter, Berlin - New York, 1983 * pages 22-25 *	1	
A	US-A-2 543 407 (HILL et al.) * claim 1 *	9	
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			C 22 B 26/10

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	11-07-1989	SUTOR W

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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EPO FORM 1503 03.82 (P0401)