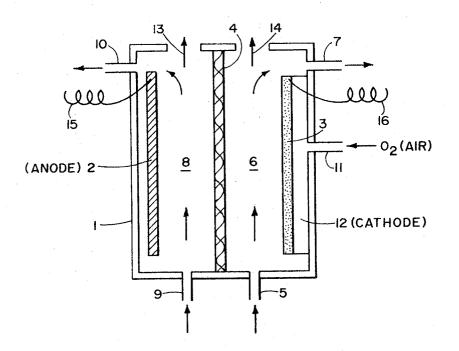
D. H. GRANGAARD 3,462,351 PROCESS FOR ALKALINE PEROXIDE SOLUTION PRODUCTION INCLUDING ALKALI CONCENTRATION CONTROL Filed Jan. 30, 1967 Aug. 19, 1969



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- 3,462,351 PROCESS FOR ALKALINE PEROXIDE SOLUTION PRODUCTION INCLUDING ALKALI CONCEN-TRATION CONTROL Donald H. Grangaard
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5 Claims

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ABSTRACT OF THE DISCLOSURE

The manufacture of a peroxide containing solution at a controlled and usually low alkalinity by an electrolytic 15 procedure in which the anolyte is provided at a higher alkali concentration than the catholyte.

BACKGROUND OF THE INVENTION Field of the invention

This invention is directed to improvements in the preparation of peroxide bleach solutions through the electrochemical reduction of oxygen. Specifically, the invention is 25 directed to exercising control over the alkali concentration of the caustic solution commonly employed in such cells.

Summary of the invention

In brief, particular objects of this invention include the attainment of greater cell efficiency (i.e., gms. or lbs. peroxide/hour) through the medium of higher electrical currents while maintaining the caustic concentration of the cathode solution relatively low. In fact, the alkali con- 35 centration of the cathode solution may be decreased while providing at least the same current flow if the principles of the invention are followed. These foregoing and other objects, I have found, may be accomplished by cycling through the anode compartment of the electrolytic cell a 40 caustic solution having a concentration which is substantially higher than the concentration of the caustic solution passed through the cathode compartment. The solution passed through the anode compartment may have an alkali concentration which is two to five times the 45 concentration of the solution passed through the cathode compartment. This facilitates the attainment of the above noted objects.

BRIEF DESCRIPTION OF THE DRAWING

The invention will be more fully understood by reference to the following detailed description and accompanying drawing wherein the single figure illustrates diagrammatically one embodiment of a single cell arrangement having electrolyte flow paths in accordance with the 55 invention.

PREFERRED EMBODIMENTS OF THE

INVENTION

60 Referring to the drawings, the numeral 1 designates the electrically non-conducting casing of an electrolytic cell having an anode of nickel designated at 2 and a porous activated carbon cathode 3. A diaphragm 4 of asbestos separates the anode from the cathode in well 65 spaced relation and inhibits the passage of the anolyte into the catholyte and vice versa. An inlet 5 to the cathode compartment 6 provides for introducing an alkaline electrolyte (the catholyte) to the cathode compartment 6. An outlet from this compartment is designated at 7 and 70 provides for the passage outwardly of the alkaline peroxide solution. The anode compartment 8 is supplied through

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an inlet 9 with an alkaline solution (the anolyte) and an outlet port is provided at 10. This analyte may be recirculated and, due to the provision of the separate compartments 6 and 8, the concentration of alkali in the anolyte may differ materially form the concentration of the alkali in the catholyte. The anolyte and catholyte flow are requested so that the electrolyte flow is outwardly from ports 7 and 10 and overflow of the cell does not occur. In the schematic diagram shown for the cell, the necessary oxygen containing gas is introduced through the inlet 11 into a chamber 12 serving basically as a manifold for feeding the gas to the porous cathode 3. Chamber 12 is itself sealed from the electrolyte in any convenient manner, as by the contact of the cathode with the casing 1.

The peroxide is formed at the cathode according to the following equation:

$O_2+H_2O+2e \Longrightarrow OH^-+HO_2^-$

The perhydroxyl then is carried from the cell with the electrolyte, electrolyte flow being indicated by the arrows 20 in the drawing. Also, arrows at 13, 14 indicate the flow of gases involved in the action from the cell which at the top is open to the atmosshere, for example.

Specifically, I have found then that, if the alkali concentration of the anolyte is 2 to 5 times the concentration of the alkali in the catholyte and voltage (2 volts) is applied between anode lead 15 and cathode lead 16, vastly improved cell efficiency in terms of grams of peroxide produced per hour can be obtained without, in turn, appreciably increasing the alkali concentration re-30 quired in the catholyte. This is extremely important since, if the peroxide is to be used for bleaching purposes, the ratio of peroxide to caustic must be held within rather narrow limits. Also, it is quite surprising that such occurs so readily and efficiently since there is electrical conduction or ionic movement, as noted hereinafter, from the compartment 8 of higher alkali concentration to the compartment 6 wherein the reaction producing the perhydroxyl takes place.

The improvements in performance obtained through operating a number of different cell designs in this manner is shown in the following table.

45	Test	Cone. of eaustic anode com- partment, percent	Sol'n supply to cathode compart., percent	Volt- age	Amperage	Gms./H ₂ O ₂ per hr.
50	A	2	2	2	3.12	1, 260 1, 520
	в	5 1	$2 \\ 1 \\ 1$	22	3.93 1.86	1. 520 0. 62 1. 15
	c	52	12	2	3.47 1.35	0.647
	D	5 2 5	$1 \\ 2 \\ 1$	$\frac{2}{2}$	1.43 3.77 4.26	1.434 1.653

The alkali employed in these specific examples was sodium hydroxide. As is known in the art, alkalis, particularly potassium hydroxide and sodium carbonate, are useful for the same purpose in generally the same concentrations.

As will be noted from the table above, a very significant increase in current and rate of peroxide production occurs simply by increasing the alkali concentration of the anode compartment. Further, the concentration of the cathode compartment alkali may be beneficially decreased if the anolyte concentration is relatively high. Thus, tests A and B above indicate that by simply increasing the anolyte concentration to 5%, the cell current and production are increased materially. Tests C and D show that the reduction of catholyte concentration, while increasing the anolyte, is also beneficial.

In these tests the flow rate of the alkali through each compartment is about the same. Flow rate through the

producing compartment 6 is limited in known manner by the fact that the flow must be sufficiently slow that adequate production of the perhydroxyl ion takes place and sufficiently fast that a reasonable quantity of solution containing the ion is removed from the cell.

The actual cell construction employed may be in general accordance with the cell structure shown in my corresponding application, co-filled herewith entitled "Process for Producing Peroxide and Electrolytic Cell Therefor," Ser. No. 612,515, filed Jan. 30, 1967.

Ser. No. 612,515, filed Jan. 30, 1967. 10 The alkaline solution passed through the inlet **5** and, augmented by the peroxide, exits through outlet **7** and is passed to a bleaching bath or to other usage or storage. A slight increase in alkalinity of the solution is occasioned by cell operation. The alkaline solution through inlet **9** is 15 decreased somewhat in alkali concentration due to movement of the cations through the diaphragm **4** towards the cathode compartment. The solution exiting through outlet port **10** thus needs only to be refortified to bring it to the original strength and it may be recirculated through 20 the same cell. If desired in battery operation, the anolyte may be passed from cell to cell.

Data indicate that in usual operation the 5% anolyte solution exits from the outlet port 10 at a concentration of about 4.7%. This may vary somewhat with flow rates 25 through the compartment and with the specific nature of the anolyte and electrolyte.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that I do 30 not limit myself to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. In a process for the manufacture of an alkaline peroxide solution which includes electrolyzing an aqueous 35 solution of an alkali which is in electrical contact with an anode and a gas porous catalytic cathode and to the surface of which catalytic cathode an oxygen containing gas is fed, the steps of passing the aqueous electrolyte

separately through anode and cathode cell compartments, and providing the solution passed through the anode compartment at a materially greater concentration than the solution passed through the cathode compartment, and removing said alkaline peroxide solution from said cathode compartment.

2. A process according to claim 1 wherein the anolyte concentration is between about two to five times the catholyte concentration.

3. A process according to claim 1 wherein the concentration of alkali passed through the cathode compartment is between about 1 to 2% by weight and the concentration of the alkali passed through the anode compartment is between about 2 to 5% by weight.

4. A process according to claim 1 wherein the volume rate of flow of the anolyte and the catholyte is substantially the same.

5. A process according to claim 4 wherein the anolyte and the catholyte are each selected from the group con-) sisting of aqueous solutions of sodium hydroxide, potassium hydroxide and sodium carbonate.

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