

Jan. 20, 1942.

E. T. LADD

2,270,376

PROCESS OF TREATING ALKALI METAL HYDROXIDE SOLUTIONS

Filed Oct. 23, 1937

Fig. 1.

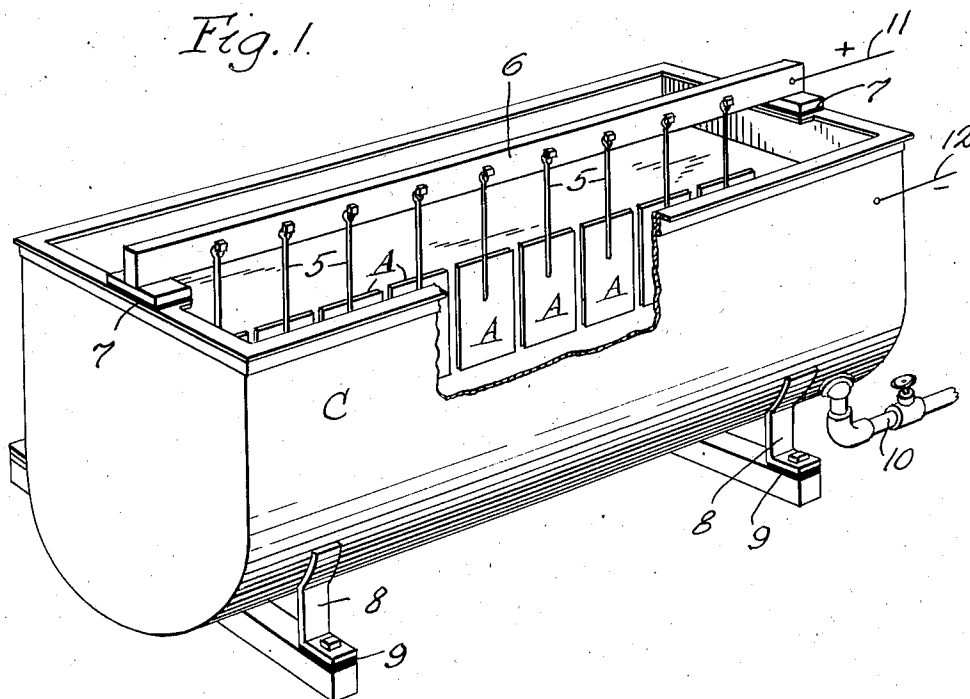
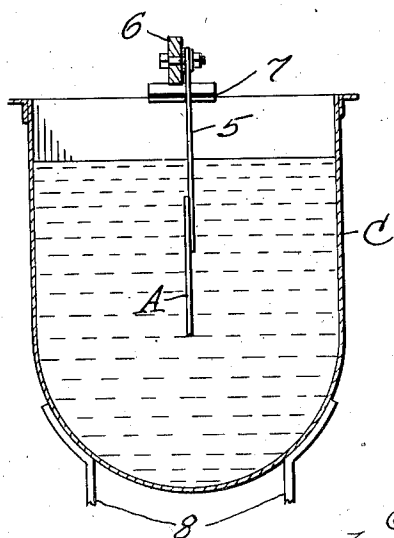


Fig. 2.

INVENTOR.

Edward Thorndike Ladd.
By Parker, Rocknow & Farmer.
ATTORNEYS.

UNITED STATES PATENT OFFICE

2,270,376

PROCESS OF TREATING ALKALI METAL
HYDROXIDE SOLUTIONSEdward Thorndike Ladd, Lewiston Heights, N. Y.,
assignor to Innis, Speiden & Co., Niagara Falls,
N. Y.

Application October 23, 1937, Serial No. 170,586

12 Claims. (Cl. 204—153)

This invention relates to improvements in purifying alkali metal hydroxide solutions.

When sodium or potassium hydroxide is produced by electrolysis of the corresponding chloride, and is subsequently concentrated by evaporation in the usual manner, the caustic solutions contact with iron or other metallic surfaces and consequently small quantities of ferric or other metal compounds are formed, some of which are soluble in the caustic solutions and are consequently difficult to remove. The presence of even small quantities of iron or other metal compounds in caustic solutions is very objectionable when these solutions are used for certain purposes.

Alkali metal hydroxide solutions when produced by electrolysis of the corresponding chlorides also generally contain some chlorine compounds, such as the chloride, hypochlorite, or chlorate, of the corresponding alkali metal, and for certain purposes, these chlorine compounds are objectionable in the alkali metal hydroxides.

One of the objects of this invention is to provide an improved process of purifying alkali metal hydroxide solutions for removing impurities in the form of metal compounds therefrom. Another object of this invention is to provide a process for removing chlorine compounds from alkali metal hydroxide solutions. It is also an object of this invention to provide a process whereby metallic impurities and chlorine compounds may be simultaneously removed from alkali metal hydroxide solutions. A further object of this invention is to provide an improved process of purifying alkali metal hydroxide solutions by subjecting the same to electrolysis. Other objects of this invention will appear from the following description and claims.

In the accompanying drawing:

Fig. 1 is a perspective view of an electrolytic cell by means of which the process embodying this invention may be carried out.

Fig. 2 is a transverse sectional elevation thereof.

In accordance with my process, I subject an alkali metal hydroxide solution to electrolysis for a period of time under conditions which permit the removal of impurities in the form of metal compounds from the solutions by depositing the metal to be removed on the cathode of the electrolytic cell. This electrolytic action may be carried out in any suitable or well known form of electrolytic cell and in the accompanying drawing, I have illustrated by way of example a cell which may be used for this purpose, it being un-

derstood, however, that it is not intended to limit this invention for use in connection with the particular cell shown. This cell may be in the form of a vessel C, in which the solution may be contained while subjected to electrolysis. The vessel C is preferably made of iron or other suitable current conducting material and forms the cathode of the electrolytic cell. The anode may be in the form of one or more plates A of any suitable current conducting material which does not become decomposed by the electrolytic action taking place within the cell. Platinum, for example, may be used for this purpose, and in the construction shown a plurality of sheets of platinum A are suspended by means of suitable wires 5 from a bus bar 6 which may be of copper or other current conducting material, and which is arranged above the top of the tank C. The ends of the bus bars 6 are, of course, insulated from the tank C in any suitable manner for example by means of insulating plates or devices 7 arranged at opposite ends of the tank and upon which the bus bar is supported. The tank may have a substantially semi-cylindrical bottom, so that all parts of the tank will be equally spaced from the anode plates A. The tank or vessel C may be suitably mounted in any desired manner and in the construction shown, is provided with feet or legs 8 which preferably rest on suitable insulating blocks 9. 10 represents a valved pipe or conduit by means of which material may be admitted to or discharged from the tank or container C.

In the carrying out of my process, a quantity of alkali metal hydroxide solution may be placed into the tank as indicated in Fig. 2 and current may be supplied to the cell by suitable conductors 11 and 12. The positive conductor 11 is connected to the bus bar 6 on which the anode plates are suspended and the negative conductor connects with the cathode or vessel C.

Current is supplied to the electrolytic cell at a voltage in excess of the polarization voltage of the iron or other metal compounds which are contained as impurities in the alkali metal hydroxide solution. Generally in caustic soda or potash solutions, iron is present in the forms of sodium or potassium ferrate or ferrite, which are soluble in concentrated solutions of caustic potash or soda. The fact that these materials are soluble has heretofore made it difficult to remove them economically from the hydroxide solutions. In addition to iron compounds, other metallic impurities equally well may be removed from the alkali metal hydroxide solutions.

Among the metallic impurities which are sometimes found in hydroxide solutions and which may be removed by my process are aluminum and manganese compounds.

In accordance with my invention, the metal compounds present as impurities in the caustic potash or soda solutions are removed by a deposit of the iron or other metal on the cathode or vessel C and by the liberation of oxygen at the anode plates. The reaction which takes place in the electrolytic cell is probably a double one due to the fact that the voltage necessary for the decomposition of the metal compounds to be removed is higher than the polarization voltage of caustic potash or soda in solution. This fact probably results, in the case of iron compounds, in a reaction in which the potassium or sodium ferrate or ferrite is initially broken up into potassium or sodium, liberated at the cathode and ferric oxide which takes on a negative charge. The alkali metal immediately reacts with the water in the solution to form alkali metal hydroxide. This negative charge of the ferric oxide is changed to a positive charge by hydrogen or oxygen released through the disassociation of caustic soda or potash, and this causes the iron to be deposited at the cathode after this oxidation has taken place. Similar reactions probably take place when other metal compounds are present as impurities in alkali metal hydroxide solutions under treatment according to my process. Regardless of theories as to the nature of the reaction, it has been found by actual use of this invention that iron and other metals present in the solution as impurities are deposited upon the cathode and that after a period of treatment, the caustic solution can be freed of all detectable traces of iron. The electrolytic action also has the property of preventing the hydroxide solution from attacking the iron of the vessel or cathode.

During the treatment of the alkali metal hydroxide solution in the electrolytic cell to remove metallic impurities from the solution, another reaction takes place which also helps to purify the solution. Because of the nature of the electrolytic cells in which the brine solutions are broken up by electrolysis to produce alkali metal hydroxides, some chlorine compounds are generally present in the hydroxide solutions. These compounds may be in the form of chlorides or hypochlorites of the alkali metal, which are readily soluble in the alkali metal hydroxide solutions, and consequently difficult to remove. I have found that by treating alkali metal hydroxide solutions in accordance with this process, any chlorides and hypochlorites of the alkali metal are converted into chlorates, probably due to the presence of nascent oxygen resulting from the decomposition of the metallic impurities or the alkali metal hydroxide solution. The chlorates may also be changed into perchlorates. Both of these chlorates are only very slightly soluble in an alkali metal hydroxide solution at room temperatures, and consequently, will precipitate or settle out of the solution at normal atmospheric temperatures to a very large degree and can be practically completely removed from the solution by moderately cooling the alkali metal hydroxide solution slightly below room temperatures, for example, to about 50° F. The alkali metal hydroxide solution can then be drawn off from the precipitate or otherwise separated from the insoluble chlorates.

During electrolysis of the alkali metal hydrox-

ide solutions in accordance with this process, a considerable amount of water is decomposed and consequently my improved process also results in a concentration of the solution to some extent.

As has been stated, the voltages and current densities under which my improved process may be carried out may vary considerably. By way of example, if the tank C be made of a size to contain approximately 900 gallons of alkali metal hydroxide solution, by subjecting the solution in the electrolytic cell shown to a current of about seven volts, it is found that a current density at the anode of about .625 ampere per square inch results. Under such conditions, if the electrolysis is carried out for a period of about forty-eight hours, the iron or other metal compounds present in the solution as impurities are removed to such an extent that no traces of them can be found by analysis, and furthermore, the chlorine compounds are to a large measure converted into insoluble chlorates which can be separated from the solution by crystallization. Furthermore, the electrolysis results in a concentration of the solution by approximately 4%. It is, of course, not intended to limit this invention to the particular example given, since wide variations in voltage and current density can be employed so long as the voltage employed is at least equal to the polarization voltage of the metal compounds which are to be removed from the solution.

The term "metallic impurities" as herein used is intended to include such metal compounds other than alkali metal compounds which may be present in alkali metal hydroxide solutions, particularly compounds of iron, manganese and aluminum.

I claim as my invention:

1. A process of removing dissolved chlorine compounds from an alkali metal hydroxide solution containing the same, which includes subjecting said solution to electrolysis at a voltage in excess of the polarization voltage of the alkali metal hydroxide solution to liberate oxygen, reacting on said chlorine compounds with said oxygen to form chlorates which are substantially insoluble in said solution, and separating said chlorates from said solution.

2. A process of removing from an alkali metal hydroxide solution chlorides and hypochlorites dissolved therein, which includes subjecting said solution to electrolysis to liberate oxygen, reacting on said soluble chlorides and hypochlorites with said oxygen to produce chlorates which are substantially insoluble in said solution and removing the crystallized chlorates from said solution.

3. A process of removing dissolved chlorine compounds from an alkali metal hydroxide solution containing the same, which includes subjecting said solution to electrolysis to liberate oxygen, reacting on said chlorine compounds with said oxygen to form chlorates, cooling the solution to cause crystallization of said chlorates, and separating said chlorates from said solution.

4. A process of purifying alkali metal hydroxide solutions which includes subjecting a solution of alkali metal hydroxide containing metallic impurities and chlorine compounds to electrolysis at a voltage in excess of the polarization voltage of the alkali metal hydroxide solution, depositing metallic impurities on the cathode and liberating oxygen at the anode; reacting on chlorides and hypochlorites contained in said solution with said oxygen to form chlorates which are substantially insoluble in said hydroxide solution, and

separating said undissolved chlorates from said solution.

5. A process of removing metallic impurities and chlorine compounds from an alkali metal hydroxide solution, which includes subjecting said solution to electrolysis at a voltage in excess of the polarization voltage of the alkali metal hydroxide solution, depositing the metal constituents of said impurities on the cathode, liberating oxygen by said electrolysis, oxidizing said chlorine compounds to form substantially insoluble chlorates, and separating said chlorates from said solution.

6. A process of removing metallic impurities and chlorine compounds from an alkali metal hydroxide solution, which includes subjecting said solution to electrolysis at a voltage in excess of the polarization voltage of the alkali metal hydroxide solution, depositing the metal constituents of said impurities on the cathode, liberating oxygen by said electrolysis, oxidizing said chlorine compounds to form chlorates, cooling said solution to crystallize said chlorates, and separating said chlorates from said solution.

7. A process of purifying an alkali metal hydroxide solution, which comprises subjecting said solution to electrolysis by introducing an electric current into said solution through an anode which is unattacked by said solution during electrolysis and at a voltage sufficient to decompose water, liberating nascent oxygen at said anode during the electrolysis, and reacting on dissolved impurities in said solution with said oxygen to form substantially insoluble crystalline compounds, and removing undissolved crystalline compounds from said solution.

8. A process of purifying and concentrating alkali metal hydroxide solutions which includes subjecting a solution of an alkali metal hydroxide containing alkali metal ferrites and ferrates and chlorine compounds in solution to electrolysis at a voltage at least equal to the polarization voltage of said iron compounds by introducing an electric current into said solution through an anode which is unattacked by said solution during electrolysis, depositing iron on the cathode, decomposing water of said solution and discharging hydrogen and oxygen from the electrodes, reacting on chlorides and hypochlorites contained in said solution with some of the oxygen resulting from said decomposition of water to form chlorates which are substantially insoluble in said hydroxide solution, and separating undissolved crystalline chlorates from said solution.

9. A process of removing alkali metal compounds of metallic impurities electrolytically

from solutions of alkali metal hydroxides, said process including subjecting the solution to electrolysis at a voltage in excess of the polarization voltage of the alkali metal hydroxide solution, electrolytically depositing on the cathode the alkali metal of alkali metal compounds of said metallic impurities, to reduce said impurities to the oxides of said metal impurities, changing the negative charge of said impurities to a positive charge, and depositing the metal impurities electrolytically on the cathode.

10. A process of removing alkali metal compounds of metallic impurities electrolytically from solutions of alkali metal hydroxides, said process including subjecting the solution to electrolysis at a voltage in excess of the polarization voltage of the alkali metal hydroxide solution, electrolytically depositing on the cathode the alkali metal of alkali metal compounds of said metallic impurities, to reduce said impurities to the oxides of the metal impurities, simultaneously liberating hydrogen gas and oxygen gas from said hydroxide solution, reacting on said metallic oxide impurities with a liberated gas to change the negative charge of said oxide to a positive charge, and depositing the metal of said positively charged metal oxide impurity at the cathode.

11. A process of removing alkali metal ferrates and ferrites electrolytically from solutions of alkali metal hydroxides, including subjecting an alkali metal hydroxide solution to electrolysis at a voltage in excess of the polarization voltage of the alkali metal hydroxide solution, electrolytically breaking up the alkali metal ferrates and ferrites by depositing the alkali metal of said ferrates and ferrites at the cathode, forming a hydroxide of said alkali metal at the cathode, changing the polarity of the iron oxides resulting from said deposit of said alkali metal on said cathode from a negative charge to a positive charge, and depositing iron from said oxides electrolytically on the cathode.

12. A method of treating an alkali metal hydroxide solution produced by electrolysis of an alkali metal salt solution and containing chloride impurities, which method includes subjecting said alkali metal hydroxide solution to electrolysis at a voltage sufficient to decompose water, reacting on chloride impurities contained in said solution with oxygen produced by electrolysis to form chlorine compounds which are substantially insoluble in said solution, said solution being concentrated by the decomposition of water of said solution.

EDWARD THORNDIKE LADD.