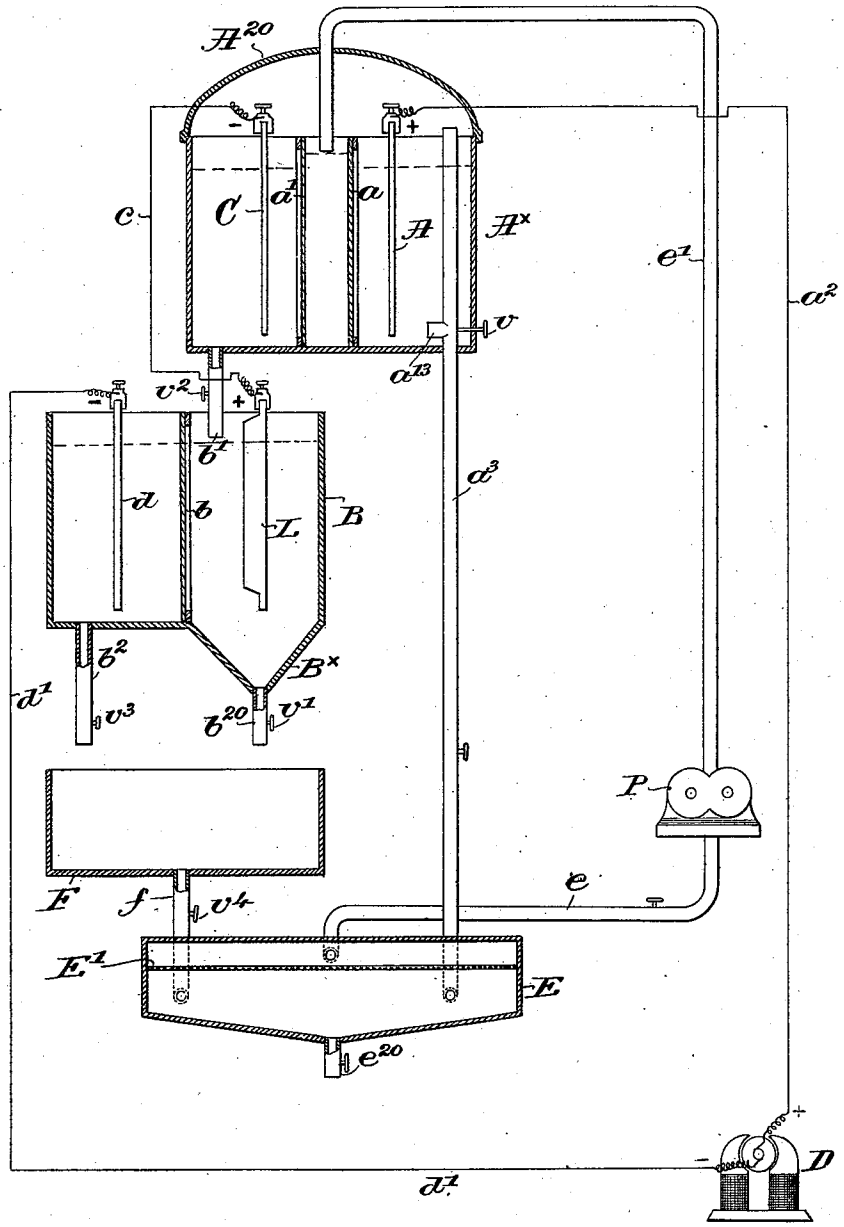
(No Model.)

A. B. BROWNE & E. D. CHAPLIN.

PROCESS OF MANUFACTURING OXIDS OF LEAD.

No. 563,554. Patented July 7, 1896.



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UNITED STATES PATENT OFFICE.

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PROCESS OF MANUFACTURING OXIDS OF LEAD.

SPECIFICATION forming part of Letters Patent No. 563,554, dated July 7, 1896.

Application filed January 28, 1895. Serial No. 536,463. (No specimens.)

To all whom it may concern:

Be it known that we, ARTHUR BENJ. BROWNE, of Cambridge, and EDWIN D. CHAPLIN, of Natick, county of Middlesex, and State of Massachusetts, have invented an Improvement in the Manufacture of Oxids of Lead, of which the following description, in connection with the accompanying drawing, is a specification, like letters on the drawing representing like parts.

This invention has for its object the production of oxids of lead in a cheap and expeditious manner by decomposing a metallic-lead anode by a current of electricity in an alkaline hydrate to form an oxid of lead, such, for instance, as the brown oxid of lead, subsequent dissolving of the oxid so produced in an alkaline hydrate forming a plumbate of an alkaline base, the neutralizing of such solution precipitating a hydrated or a carbonated oxid of lead, according to the character of the neutralizing agent employed. The alkaline hydrate is preferably obtained by the electrolytic separation of a solution of a salt of an alkaline base into an acid and an alkaline hydrate, the metallic lead as an anode being decomposed by a current of electricity in the alkaline hydrate, the oxid of lead so formed being withdrawn and subsequently treated according to the desired oxid of lead to be produced, while the acid and the alkaline hydrate are subsequently recombined to form substantially the original solution in fit condition to be used over again.

The nitrate, sulfate, or chlorid of sodium, or, in fact, any solution of a salt of an alkaline base which will separate by electrolysis into an acid and an alkaline hydrate, may be employed in carrying out our invention, and preferably such separation is maintained by interposing two foraminous diaphragms between the poles of the electric circuit and maintaining a preponderance of pressure of the electrolyte upon the inner sides of said diaphragms during the electrolytic action, whereby the acid is maintained separated outside of one diaphragm and the alkaline hydrate outside of the other, as in another application, Serial No. 532,394, filed by us December 20, 1894, wherein such process of maintaining the separation of the anion and cathion is broadly claimed.

The drawing represents diagrammatically and in vertical section a convenient form of apparatus for carrying out our invention.

A preferably hooded or closed tank or electrolyzer $A^{\times}$ is provided with two foraminous diaphragms $a\ a'$, through which the electrolyte can pass, and dividing the tank into three compartments, the anode A and cathode C being placed in the two outer compartments, respectively, and of any suitable conducting material not affected materially by the anion and cation. The anode A is connected by a wire a^2 with the positive pole of a dynamo D or other source of electrical energy. A pipe a^3, opening at its upper end into the hood A^{20}, enters the lower portion of a tank E, preferably at a considerably lower level than the tank $A^{\times}$, and said pipe is provided with an inlet a^{13} at the lower portion of the anode-compartment of the electrolyzer, controlled by a valve v.

Below the tank $A^{\times}$, but above the tank E, we place a tank B, preferably divided into two compartments by a foraminous diaphragm b, the diaphragms in both of the tanks $A^{\times}$ and B being preferably canvas or light textile material. One of the compartments has a hopper-shaped bottom $B^{\times}$, provided with a discharge-pipe b^{20}, controlled by a valve v', and said compartment is connected by a pipe b' with the cathode-compartment of the tank $A^{\times}$, and it is provided with a suitable valve v^2, and in this compartment of tank B is placed the metallic lead L to be acted upon, preferably in pig form, and forming the anode of said tank, connected electrically by wire c with the cathode C of the tank $A^{\times}$. A cathode d of suitable conducting material is placed in the second compartment of the tank B, and it is connected by wire d' with the negative pole of the dynamo D or other source of electrical power. A pipe b^2, provided with a valve v^3, discharges the contents of the cathode-compartment into a tank F, intermediate the tanks B and E, said tank F having a discharge-pipe f opening in

the lower part of tank E, the flow being controlled by a valve v^4, while the suction-pipe e of the pump P draws the fluid contents of said tank E from its upper portion, and by means of the pump discharge or delivery pipe e' said contents are emptied into the tank $A^\times$ between the two foraminous diaphragms a and a'.

Supposing now that the electric current passes through the electrolyte in the tank $A^\times$ from the anode to the cathode, and in the tank B through the fluid therein from the metallic lead L to the cathode d, the electrolyte in the tank $A^\times$, which will be a solution of a salt of an alkaline base, as, for instance, a solution of nitrate of soda, is introduced as fast as needed by pipe e' between the diaphragms a and a', and the pressure thereof upon the inner sides of the diaphragms is the greater by maintaining the level of the electrolyte therebetween higher than the level of the liquids in the two outer compartments. The passage of the electric current through the electrolyte separates the latter into nitric acid outside of the diaphragm a and into sodium hydrate outside of the diaphragm a', and such separation is maintained by the said preponderance of pressure upon the inner sides of the diaphragms. The alkaline hydrate flows through the pipe b' into the compartment of the tank B containing the metallic lead, and during the electrolytic action said metallic lead is decomposed and forms brown oxid of lead, which settles into the hopper-like bottom $B^\times$, from which it may be withdrawn through the discharge-duct b^{20}.

If the brown oxid of lead is desired, it is withdrawn directly for subsequent use, but if other oxids of lead are to be produced the said brown oxid is further treated, and to facilitate such treatment it is conveyed from the tank B to the tank or vat F.

While the metallic lead has been transformed into brown oxid of lead, the alkaline hydrate will have been passing through the diaphragm b to the cathode d in the tank B, and if it is withdrawn therefrom by the pipe b^2 and discharged into the tank F it will dissolve the brown oxid of lead and form a plumbate of an alkaline base, in this instance plumbate of soda. The plumbate of soda so formed and in solution is then withdrawn by pipe f and discharged into the tank E, which latter is provided with a suitable filter E', the plumbate of soda entering below the filter, as shown in the drawing. By neutralizing the solution in the tank E lead hydrate will be precipitated, the neutralization being effected by adding an acid thereto. In the present instance the nitric acid separated in the electrolyzer $A^\times$ is employed as the neutralizing agent. The nitric acid is drawn from tank $A^\times$, through tank a^3, into tank E, the valve v of the inlet a^{13} having been opened, and it is mixed with the plumbate of soda in solution, neutralizing said solution and precipitating hydrated oxid of lead, which settles to the hopper-like bottom of the tank E, and it is removed from time to time by means of a suitable gate e^{20}. The remaining solution in the tank E, from which the hydrated oxid of lead has been precipitated, is, as will be obvious, a solution of nitrate of soda, and fit to be used over again. It passes up through the filter E' and is pumped out of the tank E by the pump P and discharged into the electrolyzer $A^\times$, fresh electrolyte being added from time to time as necessary.

If it is desired to produce a carbonated oxid of lead instead of a hydrated oxid of lead, the solution of a plumbate of an alkaline base may be neutralized by a carbonic anhydrid, and by means of the apparatus herein shown such carbonic anhydrid may be produced in the carrying out of the process. In such instance we would use a solution of a carbonate of soda in the electrolyzer $A^\times$, such electrolyte being decomposed by the electric current into an alkaline hydrate with the liberation of carbonic anhydrid, which collects and is retained in the dome A^{20} of the electrolyzer.

The alkaline hydrate will be conducted to the tank B, as hereinbefore, the brown oxid of lead will be formed therein, and it and the alkaline hydrate which passes to the cathode-compartment in said tank will be withdrawn to the tank F to form a plumbate of an alkaline base in solution, and the solution will be discharged into the tank E, all as hereinbefore set forth. The inlet a^{13} in the pipe a^3 will then be closed, and the carbonic anhydrid will be admitted to the upper end of said pipe from the dome A^{20} and will pass into the tank E, acting therein as the neutralizing agent for the solution therein contained and precipitating a carbonated oxid of lead, which can be withdrawn from the tank, the remaining solution being a carbonate of soda in substantially its original condition and fit to be used over again in the electrolyzer, making a continuous process.

While we have shown a form of apparatus which is well adapted to carry out our process conveniently and expeditiously, other forms of apparatus may be employed, our process not being confined to any particular form of apparatus for its successful operation.

It is preferable to make the process continuous on the grounds of cheapness and expedition, but it will be obvious that the metallic lead as an anode may be decomposed in any alkaline hydrate, no matter how produced or obtained, and the plumbate of an alkaline base in solution may be neutralized by a suitable agent, no matter how obtained, provided such neutralization will precipitate an oxid of lead desired.

The production of such desired oxid of lead may be carried out by the neutralization of a plumbate of an alkaline base in solution, no matter how such solution may be obtained or produced.

We claim—

The process of manufacturing oxids of lead,

which consists in electrolytically separating a solution of a salt of an alkaline base into an alkaline hydrate, and a neutralizing agent, dissolving a metallic-lead anode by a current of electricity in such alkaline hydrate, dissolving the oxid of lead so produced by an alkaline hydrate to form a plumbate of an alkaline base, and neutralizing such solution by the electrolytically-separated neutralizing agent, to precipitate the desired oxid of lead, and collecting the remaining solution in substantially the condition of the original electrolyte, substantially as described.

In testimony whereof we have signed our names to this specification in the presence of two subscribing witnesses.

ARTHUR BENJ. BROWNE.
EDWIN D. CHAPLIN.

Witnesses:
JOHN C. EDWARDS,
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