No. 871,161. PATENTED NOV. 19, 1907.

E. D. CHAPLIN.

ART OF PRODUCING PIGMENTS BY ELECTROLYSIS.

APPLICATION FILED FEB. 3, 1906.

2 SHEETS—SHEET 1.

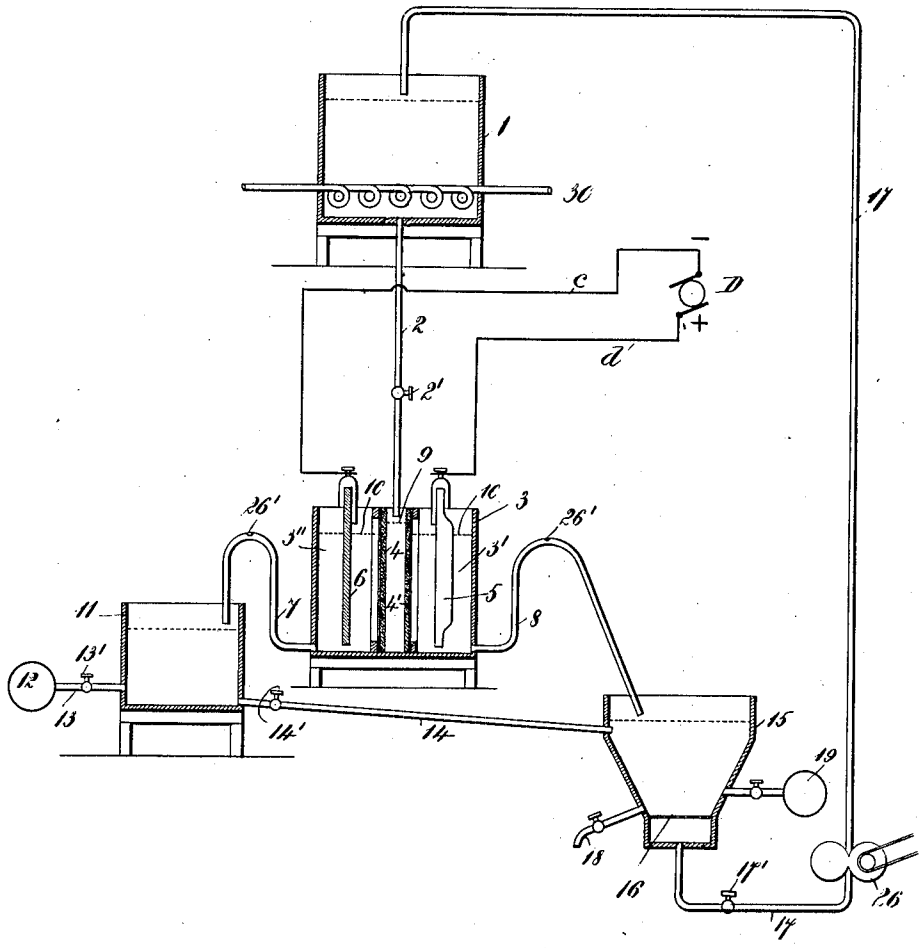


Fig. 1.

WITNESSES:
E. B. Tomlinson.
Patrick J. Conroy.

INVENTOR:
Edwin D. Chaplin
by Browne & Woodworth
his attorneys.

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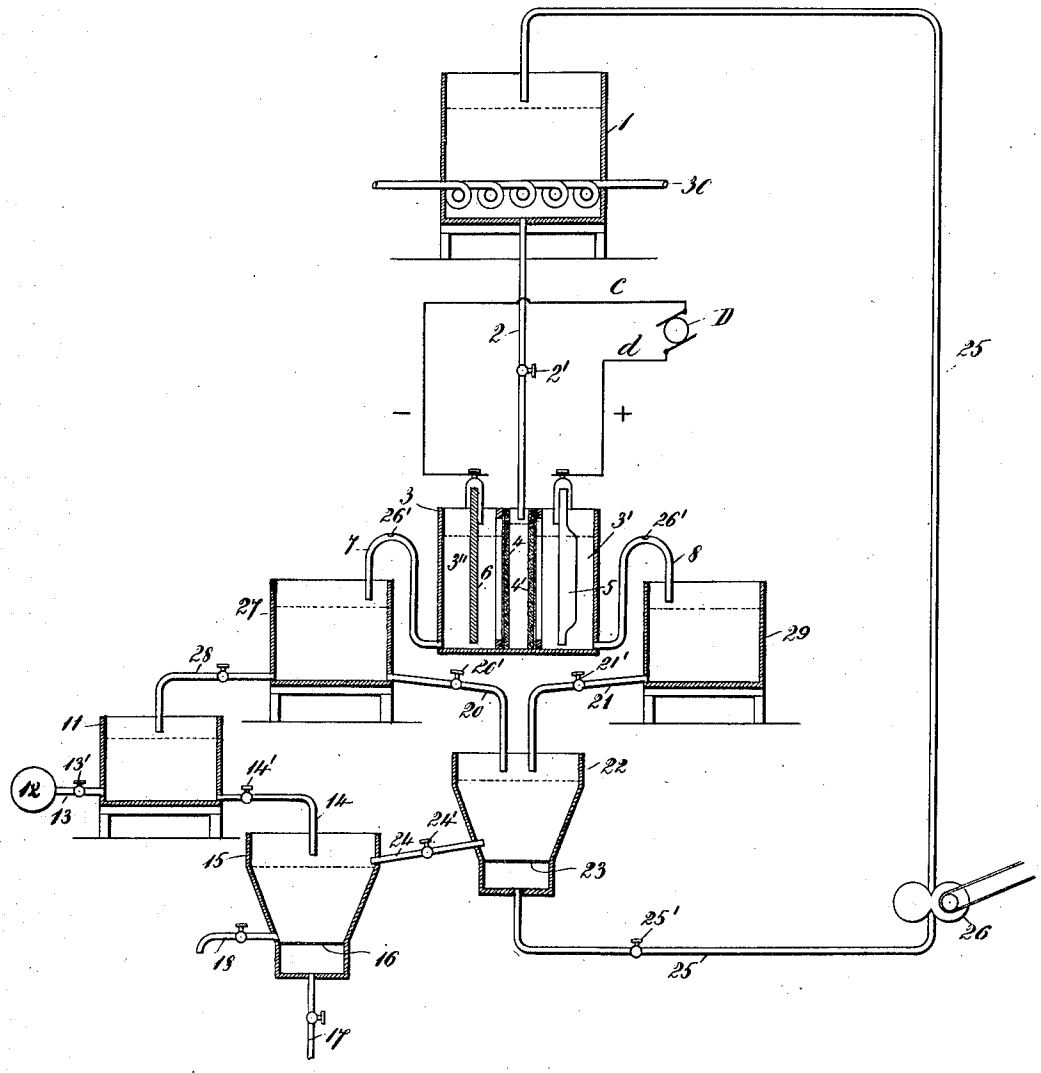


Fig. 2.

WITNESSES:
E. B. Tomlinson
Patrick J. Conroy.

INVENTOR:
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UNITED STATES PATENT OFFICE.

EDWIN D. CHAPLIN, OF BOSTON, MASSACHUSETTS, ASSIGNOR TO INTERNATIONAL LEAD COMPANIES, A VOLUNTARY ASSOCIATION.

ART OF PRODUCING PIGMENTS BY ELECTROLYSIS.

No. 871,161. Specification of Letters Patent. Patented Nov. 19, 1907.

Application filed February 3, 1906. Serial No. 299,237.

To all whom it may concern:

Be it known that I, EDWIN D. CHAPLIN, a citizen of the United States, and a resident of Boston, in the county of Suffolk and State of Massachusetts, have invented a new and useful Improvement in the Art of Producing Pigments by Electrolysis, of which the following is a specification.

My invention relates to the art of producing pigments by electrolysis, and it relates more particularly to a process for so producing white lead or lead carbonate.

In the manufacture of lead carbonate by electrolysis, I prefer to employ an electrolyte which under the influence of the electric current will separate into a solvent of lead and an alkaline hydrate and thereby to produce in the anode compartment containing metallic lead, which may be the anode of the electrolyzer, a solution of a soluble salt of lead, and in the cathode compartment, an alkaline hydrate. I have found it essential to the commercial production of white lead to maintain the lead solution and the alkaline hydrate separate and not to produce any lead precipitate in the electrolyzer itself. I have also found it essential to employ some means, such as an oxidizing agent, to prevent the formation of insoluble salts of lead in the electrolyzer.

As clearly set out in Letters Patent Nos. 555,232 dated February 25, 1896 and 563,553, dated July 7, 1896 granted to A. B. Browne and myself, and in Letters Patent No. 675,555 dated June 4, 1901 granted to H. G. Halloran and myself, certain insoluble basic salts of lead are formed in the electrolyzer, thereby rendering the processes uncommercial, unless certain precautions are taken in order to prevent such formation. According to the process described Letters Patent No. 675,555, a soluble acid salt of lead is produced by forming from metallic lead as an anode a solution of a soluble salt thereof in the presence of a soluble chlorid salt and eventually carbonating the resulting lead oxychlorid. By these operations the formation of insoluble lead salts in the electrolyzer is prevented. In Letters Patent No. 563,553, the same result is accomplished, although not so perfectly, by the addition to the electrolyzer of an oxidizing agent, specified in the patent as nitric acid. In carrying out the process set forth in Letters Patent No. 675,555, the soluble chlorid salt probably is converted into a soluble chlorate which operates in a manner analogous to the nitric acid employed in the process described in the earlier patent.

The object of the present invention is to provide a process of producing white lead by electrolysis in which the formation of insoluble salts of lead in the electrolyzer may be prevented by the use of a soluble chlorate salt which is added directly to the electrolyte instead of being formed indirectly by electrolytic action from a soluble chlorid salt.

My invention may best be understood by having reference to the drawings which accompany and form a part of this specification, and which illustrate in diagrammatic form two arrangements of apparatus and circuits whereby my process may be carried into effect; but it is to be understood that my invention is broader than mere apparatus and may be carried into effect by a great variety of apparatus and circuit arrangements.

In the drawings, Figure 1 represents an apparatus for carrying out my direct process for producing white lead and Fig. 2 represents an apparatus for carrying out my indirect process for producing white lead.

In Figs. 1 and 2, 1 represents a supply tank containing an electrolyte, which under the influence of the electric current will be separated into an alkaline hydrate and a solvent of lead, such for example as an aqueous solution of a non-alkaline salt, preferably sodium nitrate, and containing also an aqueous solution of a soluble chlorate salt which may be a chlorate of an alkaline base, preferably sodium chlorate, or any other suitable chlorate salt.

I shall hereinafter refer to the solution in tank 1 as the "neutral solution" and shall assume that it consists of sodium nitrate and sodium chlorate.

3 represents an electrolyzer divided into an anode compartment 3′ and a cathode compartment 3″, by means of the foraminous diaphragms 4 4′, between which is formed a compartment for receiving the electrolyte from the tank 1 through the pipe 2, which is provided with a valve 2′. For the purpose of preventing a mixture of the anolyte and catholyte in the electrolyzer and consequently a precipitation of lead hydrate in the electrolyzer, I may maintain a preponderance of pressure on the inner sides of said diaphragms,

preferably by maintaining the level of the electrolyte between the diaphragms higher than the level of the anolyte and catholyte. The level of the electrolyte in the compartment between the diaphragms is indicated by the dotted line 9, and the level of the anolyte and catholyte, which is governed by the height of the bends of the outlet pipes 7 and 8, is indicated by the dotted lines 10, 10. In order to prevent said outlet pipes from siphoning the liquids out of the electrolyzer, the upper portion of each of said bends may be provided with an opening 26, 26′ to equalize the atmospheric pressure on the columns of liquid in the two portions of each of said pipes.

The lead to be operated upon is placed in the anode compartment and may form the anode of the electrolyzer. In the present case the lead is shown in the form of a lead pig 5, connected by the conductor *d* to the positive pole of the dynamo D, or other suitable source of current. The cathode of the electrolyzer 6 may consist of copper or any other suitable metal, and is connected by the conductor *c* to the negative pole of the dynamo.

When the electrolyzer is charged with the neutral solution and the electric current passed therethrough, a soluble acid salt of lead is formed in the anode compartment 3′ and an alkaline hydrate, in the present case assumed above, sodium hydrate, is formed in the cathode compartment 3″

Presumably lead nitrate is at first formed in the anode compartment and this, by the continued action of the electric current, is converted into lead oxychlorid which is soluble at the temperature at which the electrolyte is maintained by the steam coil 30, or by any other suitable means. The lead oxychlorid being an acid salt of lead, renders impossible the formation of insoluble basic salts of lead.

The term "oxychlorid of lead" herein used has its usual meaning—viz., chlorid salts of lead containing oxygen in varying proportions—such, for instance as

$$PbClO_3, PbO.PbCl_2, \&c.$$

According to the indirect process which may be carried out by the apparatus shown in Fig. 1, the solution of the acid salt of lead so formed in the anode compartment 3′, is drawn off by the pipe 8 into the precipitating tank 15. The alkaline hydrate is drawn off by the pipe 7 into the tank 11, where it may be converted into an alkaline carbonate by subjecting it to the action of carbon dioxid which may be formed in any suitable manner in the generator 12, and which may be conducted into the tank 11 by means of the pipe 13 provided with a valve 13′. The alkaline carbonate so formed is drawn off through the pipe 14, provided with the valve 14′, into the precipitating tank 15 where it is mixed with the soluble lead salt and converts the latter into lead carbonate. The lead carbonate so formed settles upon the filter 16 at the bottom of the tank 15, while the filtrate which is substantially the original electrolyte, is pumped by the pump 26 through the pipe 17 provided with a valve 17′ to the supply tank 1, and may be used over again. The lead carbonate formed in the tank 15 may be drawn off through the pipe 18, washed, and run through a filter press.

Instead of precipitating lead carbonate from the solution of the soluble salt of lead which is discharged by the pipe 8 into the precipitating tank 15 by means of an alkaline carbonate, I may discharge the sodium hydrate into the tank 15 and at the same time pass carbon dioxid from the generator 19 through the solutions in said tank 15, thereby producing lead carbonate.

According to the indirect process which may be carried out by the apparatus shown in Fig. 2, the solution of the aforesaid acid salt of lead may be discharged into the settling tank 29 by the pipe 8, and thence into the precipitating tank 22 by the pipe 21, which is provided with the valve 21′, or as is obvious, it may be discharged directly into said precipitating tank.

The alkaline hydrate produced in the cathode compartment 3″ may be discharged into the settling tank 27 by the pipe 7 and thence into the precipitating tank 22 by the pipe 20 which is provided with the valve 20′, or as is obvious, it may be discharged directly into said precipitating tank.

In the tank 22, lead hydrate is precipitated from the acid salt of lead and settles on the filter 23 at the bottom of said tank while the filtrate, which in the present assumed case consists probably of a mixture of sodium nitrate, sodium chlorate and sodium chlorid, is pumped back to the supply tank 1 through the pipe 25, provided with the valve 25′, by the pump 26.

A portion of the alkaline hydrate formed in the cathode compartment 3″ is conveyed to the tank 11, either directly from the electrolyzer or from the settling tank 27 by the pipe 28 provided with the valve 28′, and is converted into a bi-carbonate of an alkaline base,—sodium bicarbonate in the present assumed case,—by treating the same with carbon dioxid produced in the generator 12, which connects with the tank 11 by the pipe 13 provided with the valve 13′.

The lead hydrate formed in the tank 22 is discharged by the pipe 24 provided with the valve 24′ into the precipitating tank 15, and the bicarbonate formed in the tank 11 is conveyed into said tank through the pipe 14 provided with the valve 14′, thereby converting the lead hydrate into lead carbonate.

The lead carbonate so formed settles at the bottom of the tank 15 on the filter 16 and may be drawn off through the pipe 18, washed, and run through a filter press. The filtrate may be drawn off by the pipe 17.

By either the direct or the indirect process above described I am enabled, by the direct employment of a soluble chlorate salt, most effectively to prevent the formation of insoluble basic salts of lead, the formation of which is fatal to a commercial production of white lead. The process is continuous and, in addition to the lead and carbon dioxid consumed, requires only the addition from time to time of fresh quantities of neutral solution.

I claim:

1. As an improvement in the art of producing white lead by electrolysis, the process herein described which consists in electrolytically dissolving metallic lead in an aqueous solution of a non-alkaline salt in the presence of a soluble chlorate salt and converting the resulting lead oxychlorid into lead carbonate.

2. As an improvement in the art of producing white lead by electrolysis, the process herein described which consists in electrolytically dissolving metallic lead in an aqueous solution of a non-alkaline salt in the presence of a soluble chlorate salt, thereby producing an alkaline hydrate and lead oxychlorid, withdrawing and mixing said lead oxychlorid and said alkaline hydrate to precipitate lead hydrate, and then carbonating said lead hydrate to produce lead carbonate.

3. As an improvement in the art of producing white lead by electrolysis, the process herein described which consists in electrolytically dissolving lead in an aqueous solution of a non-alkaline salt in the presence of a soluble chlorate salt, thereby producing lead oxychlorid, withdrawing said lead oxychlorid and converting the same into lead hydrate and then carbonating said lead hydrate to produce lead carbonate.

4. As an improvement in the art of producing white lead by electrolysis, the process herein described which consists in electrolytically dissolving lead in an aqueous solution of a non-alkaline salt in the presence of a soluble chlorate salt, thereby producing a solution of a soluble acid salt of lead, and then converting said soluble acid salt of lead into lead carbonate.

5. As an improvement in the art of producing white lead by electrolysis, the process herein described which consists in electrolytically dissolving lead in an aqueous solution of a non-alkaline salt in the presence of a soluble chlorate salt, thereby producing an alkaline hydrate and a solution of soluble acid salt of lead, withdrawing and mixing said soluble acid salt of lead and said alkaline hydrate to precipitate lead hydrate and then carbonating said lead hydrate to produce lead carbonate.

6. As an improvement in the art of producing white lead by electrolysis, the process herein described which consists in electrolytically dissolving lead in an aqueous solution of a non-alkaline salt in the presence of a soluble chlorate salt, thereby producing a solution of a soluble acid salt of lead, converting the same into lead hydrate, and then carbonating said lead hydrate to produce lead carbonate.

In testimony whereof, I have hereunto subscribed my name this fifth day of Jan. 1906.

EDWIN D. CHAPLIN.

Witnesses:

CHARLES C. KURTZ,

GEO. K. WOODWORTH.