(No Model.)

O. LUGO & H. T. JACKSON.

METHOD OF ELECTROLYTIC TREATMENT OF SOAP LYES.

No. 558,970. Patented Apr. 28, 1896.

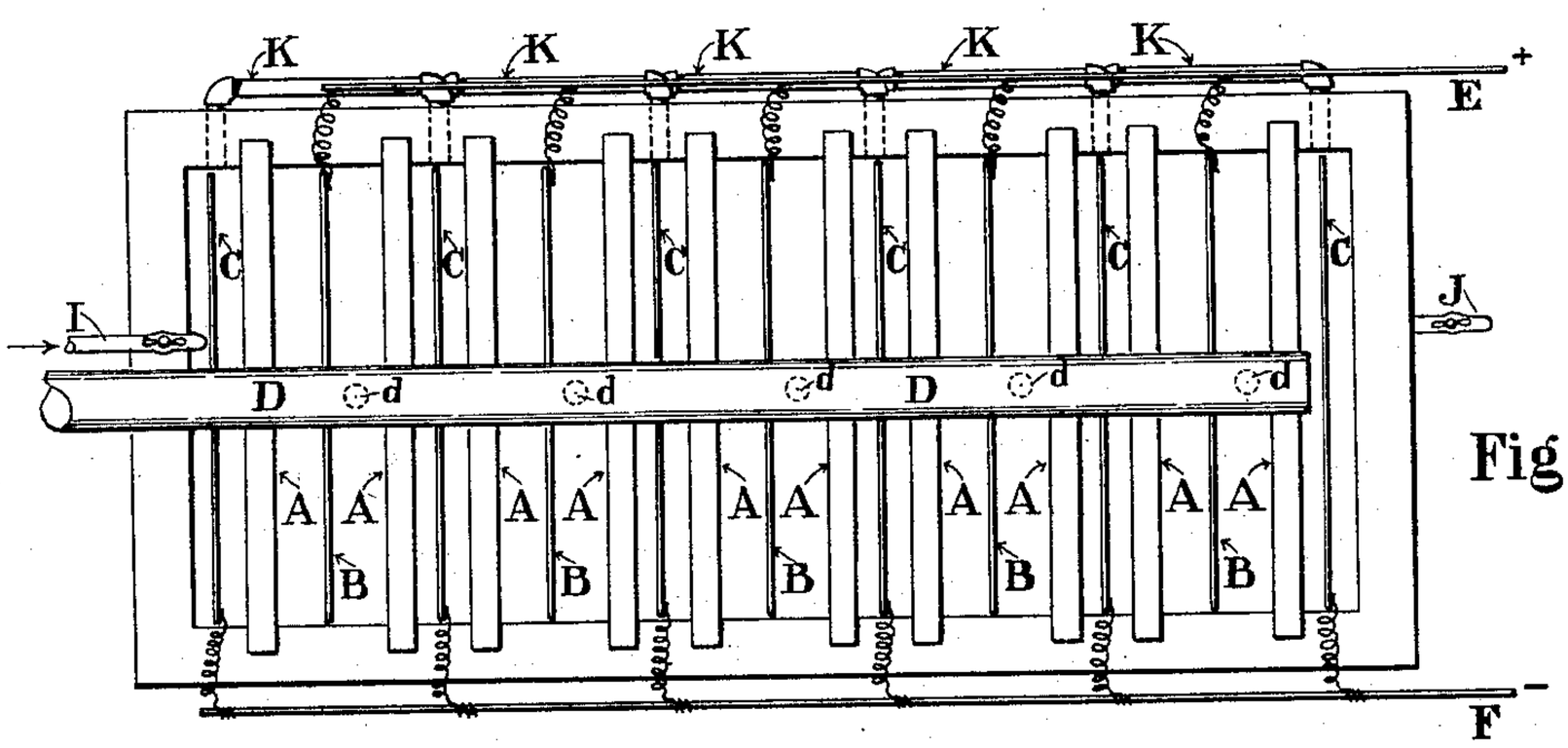

Fig. 1.

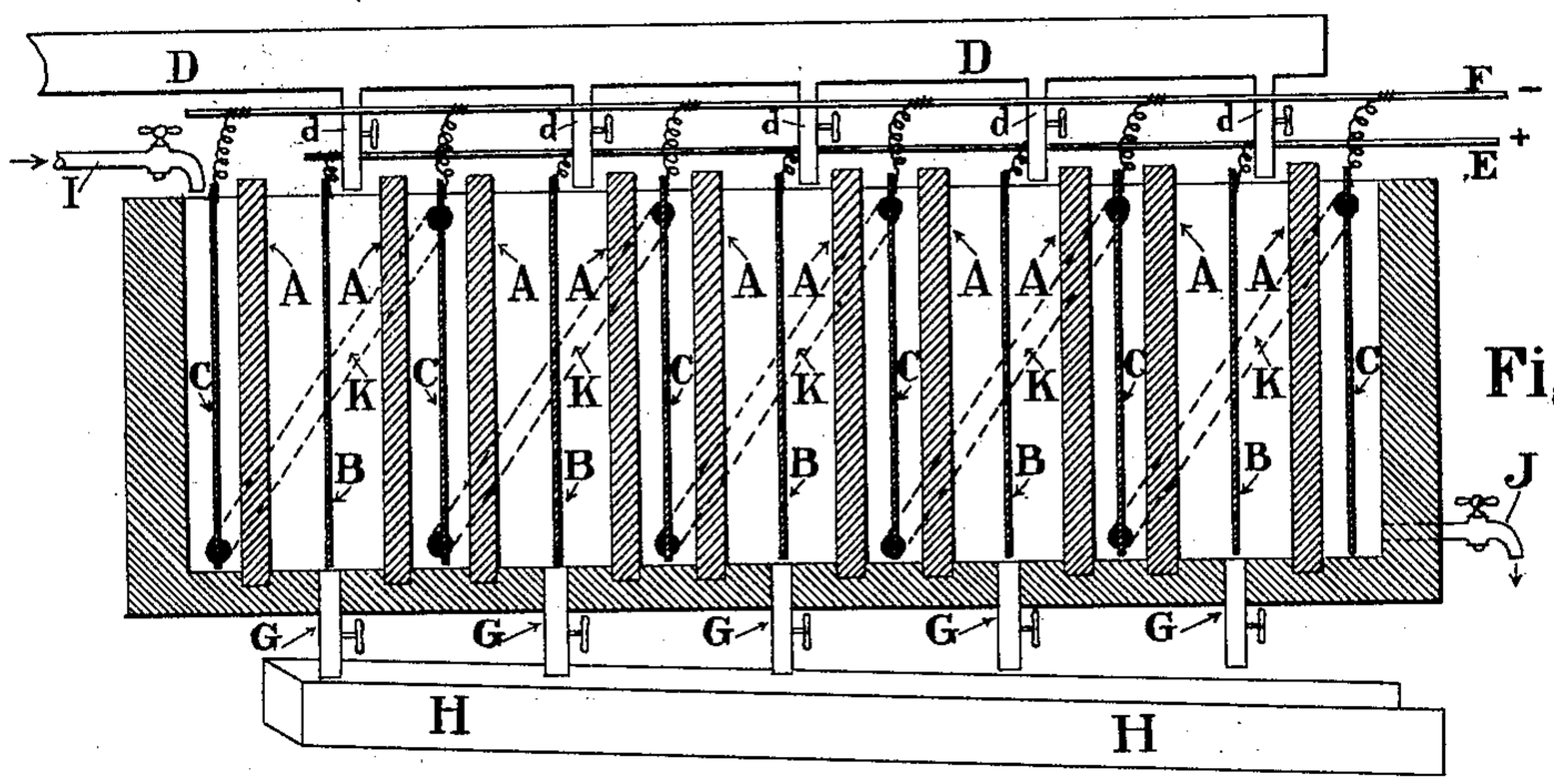

Fig. 2.

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UNITED STATES PATENT OFFICE.

ORAZIO LUGO AND HUSON T. JACKSON, OF NEW YORK, N. Y.

METHOD OF ELECTROLYTIC TREATMENT OF SOAP-LYES.

SPECIFICATION forming part of Letters Patent No. 558,970, dated April 28, 1896.

Application filed October 4, 1895. Serial No. 564,627. (No specimens.)

To all whom it may concern:

Be it known that we, ORAZIO LUGO and HUSON T. JACKSON, citizens of the United States of America, and residents of New York city, in the county and State of New York, have invented a new and useful Improvement in the Method of Electrolytic Treatment of Soap-Lyes for the Production of Glycerin and a Caustic Alkali and for the Purification of Glycerin, of which the following is a specification.

After the saponification of the fats in the manufacture of soap and the elimination of the solids formed—such as sodium or potassium oleate, $3(NaC_{18}H_{33}O_2,)$ sodium or potassium stearate, and sodium or potassium palmitate—there is remaining a liquor commonly known as "soap-lye," which contains some free alkali, sodium or potassium hydrate, some fats which are the glycerides of the fatty acids stearic, oleic, palmitic, &c., free glycerin, which was liberated by the saponification of the fats with a caustic alkali or water, some of the fatty acids themselves, some impurities, including albuminoidal matters, and from eight to ten or twelve per cent. of salt sodium chlorid, which was added to the soap solution to prevent solubility. Owing to the value of some of these constituent substances of the lye solution—namely, the glycerin, free and in combination, and the alkaline hydrate or the alkaline salts—it has been the endeavor of scientists and manufacturers to discover a method and devise an apparatus by the use of which these by-products of value could be eliminated and recovered separately and in a state of purity, to be again utilized for the manufacture of soap or put, respectively, upon the market.

The object of this invention is to extract all of the free alkali, decompose the alkaline salts with the extraction of the caustic alkali thus set free, the clarification of the lye solution (the glycerin-containing solution) by the precipitation of the albuminoidal matters and other impurities, and the decomposition of the glycerin-containing compounds, setting free a larger amount of available glycerin, obviating the use of all chemicals, rendering the solution sufficiently clear and pure that glycerin is obtained with but little discoloration after one distillation, and of such purity that it will be available for all practical uses, especially for the purpose of manufacturing nitroglycerin or "high explosives" with safety.

The further object of this invention is to render useful and of value the large amount of soap-lye which is now substantially a waste product.

It has been tried to treat this soap-lye with various reagents and then distil for the production of glycerin; but this has been accomplished with only partial success, obtaining but a part of the available glycerin, owing to the presence of impurities. These impurities are the glycerin compounds which have not been decomposed, the alkalies or alkaline salts, and the albuminoidal matters, the latter especially causing decomposition during distillation and condensation accompanied with the formation of acroleine and acrilic acid with the corresponding loss of glycerin. When the glycerin-containing solution is in this impure condition, as it is after the ordinary treatment with reagents and the subsequent filtration, impurities are carried over during distillation which materially reduce the value of the distillate and render it unfit for the production of high explosives, a purpose for which the major portion of glycerin is used. These impurities, being almost entirely in solution, cannot be eliminated by bone-black filtration and only partially by a redistillation, as they are "carried over" by the steam along with the vaporized glycerin. Bone-black filtration removes the discoloration; but often the per cent. of purity is lessened rather than increased, as the glycerin takes up other impurities contained in the bone-black. By the distillation of the lye solution in such a state of impurity a large and troublesome "foot" is left which cannot be utilized and which seriously hinders successful distillation, and, owing to some extent to chemicals used and impurities not decomposed or eliminated, crustations are formed upon the heating-tubes and apparatus which cause inefficiency of the steam used and often necessitate closing down in order to remove them. This is a serious drawback toward economical working.

The glycerin obtained from the solution

under the present mode of working is of a most crude quality and very dark in color, owing to the non-removal of the impurities.

There is a large amount of crude condensed glycerin produced which is too impure for use in the manufacture of high explosives, and heretofore the refiners have been compelled to resort to redistillations in order to obtain a product of sufficient purity for all uses. The impurities in this glycerin are albuminoidal and coloring-matters, a caustic alkali, and alkaline-glycerin compounds.

By the use of our electrolytical process the alkalies are extracted and the alkaline-glycerin compounds decomposed, with the extraction of the caustic alkali and setting free of the glycerin, which now becomes available, and the albuminoidal and coloring-matters rendered insoluble and removed by filtration. The glycerin is now of such purity that it is adapted for any use without subsequent distillations. The color is changed from a dark brown to a light straw-yellow and the glycerin rendered perfectly transparent.

By the use of this method for treating soaplyes for the purpose of distilling for the production of glycerin we obtain all the caustic alkali in the solution, with the exception of about two-thirds of that contained in the salt, NaCl, which salt is only partially decomposed, and the value of this caustic alkali obtained practically pays for the electrolytical working of the process. All of the chlorid of sodium compound could be decomposed and the caustic soda obtained without injury to the remainder of the electrolyte solution; but carrying the electrical treatment to such an excess does not prove to be of economical advantage, as all of the glycerin compounds have already been decomposed and the practically complete precipitation of the albuminoidal and coloring-matters accomplished. The solution is rendered clear and bright when sent to the still and is neutral to litmus test. The electrolytical treatment is discontinued when the neutral point is reached, when the solution will be then found clear and transparent. The distillate (glycerin) obtained from this solution will be practically pure and free from all discoloration after one distillation and be applicable for any and all purposes.

The foot left from the distillation of the lye solution after treatment by this process is composed almost entirely of salt, (sodium chlorid,) all other impurities having been eliminated, and is of such purity that it can be again used in the manufacture of soap.

It will be found that the crustations so generally found upon the heating-surfaces and upon the still or condensing apparatus will be almost entirely avoided, and the necessity of cleaning for the removal of such coatings will be practically entirely done away with. This will be found to be of great practical advantage and a material aid toward economical operation and efficiency of the amount of steam used.

The result of this electrolytical process and the changes and decompositions that take place are practically as follows: The solution containing the glycerin or the lye solution is the electrolyte and is in contact with the anode, which is composed of metallic zinc. The cathode may be of lead or iron and is immersed in water, which is separated from the electrolyte solution by means of a porous medium or diaphragm. This diaphragm and a form of appartus will be described farther on in this specification. Electrical connections are made and the constant electric current is forced to flow from the zinc anode through the electrolyte solution, the porous diaphragm, and the water to the cathode. The result follows, viz: All the free caustic soda in the electrolyte is decomposed by the action of the electric current, and the sodium is attracted to the cathode, where it immediately becomes oxidized by taking up one atom of oxygen from the water, forming oxid of sodium. This combines with one molecule of water, forming sodium hydrate or caustic soda. If potassium should be present, the action would be similar. The alkali-glycerin compounds are decomposed, being electrolytes, and the caustic alkali also found in solution at the cathode. The glycerin being set free in the electrolyte solution, compounds of a caustic alkali and a fatty acid, which have not been taken out in the manufacture of soap or saponifications, are decomposed with the extraction of the caustic alkali, as described. A part of the sodium chlorid is also decomposed with a similar result. The albuminoidal matters are precipitated and the electrolyte solution has now become perfectly clear and is neutral, or the treatment may be continued until slightly acid. It is also filled with a flocculant precipitate. The solution is now filtered in any convenient way. (A mere straining to retain the precipitate formed will be sufficient.) The filtrate containing the glycerin is bright and clear, of a slight yellow tint or perfectly water-clear, according to the time of treatment. It contains practically no impurities, except some salt sodium chlorid. From it almost perfectly pure glycerin is distilled without discoloration. The precipitate retained in the filter is composed of the impurities extracted. In one batch of lye solution treated we obtained a precipitate which contained 17.75 per cent. of albuminoids not soluble in ether or petrolether. This precipitate contained 21.73 per cent. of water. The remainder of the precipitate was sulphates, chlorids, oxids of iron, zinc, and alumina, about five per cent. of silica, and some oxy-fatty acids. The caustic alkali is practically pure and can be again used by the soap-maker. We produce by this alkali solution the density required by the soap-maker, and thus avoid condensation and the subsequent dissolving.

When a solution containing crude glycerin is submitted to electrolysis, the operation is practically the same. Caustic alkali is extracted and obtained as described, but not to such a great extent, and the amount of precipitate will depend upon the purity of the glycerin and is generally composed of albuminoids, fatty ethers, or undecomposed fats.

This invention is carried practically into effect as follows, although we do not limit ourselves to this mode of apparatus, by the use of which our invention is put into practical operation, it being evident that other forms of apparatus are equally applicable, according to the amount of solution desired to be treated. We prefer the following for work upon a large scale, as being especially adapted for the use of manufacturing caustic alkalies: An elongated tank, constructed, preferably, of brick, is divided into compartments by means of slabs of sandstone, or marble, or thin porous brick, which are the diaphragms. The number of these compartments will depend upon the amount of solution desired to be treated, or the depth and width may be increased, it being advisable to keep them comparatively narrow from diaphragm to diaphragm, in order to keep the electrical resistance as low as possible and at the same time to insure the complete treatment of the electrolyte. Each alternate compartment will contain the electrolyte and an anode of zinc, while the other series of alternate compartments will be cathode-compartments and contain water. These cathode-compartments are connected outside of the electrolytical tank by means of lead pipes extending from the bottom of the first cathode-compartment to the top of the next cathode-compartment, and so on to the end of the series of compartments. Water is allowed to flow into the first cathode-compartment and by means of these pipe connections continue to flow to the next cathode-compartment, and so on to the last cathode-cell, where it is taken out. During its passage, being in contact with the various cathodes, it has become part of the electric circuit, and will be found to be a solution of the caustic alkali which was contained in the electrolyte or soap-lye solution. The flow of water into the first cathode-compartment is regulated by a float-cock, and this float-cock is governed by the rate the solution is allowed to run from the last of this series of compartments. By this means the density of the caustic-alkali solution is so regulated that a constant stream is obtained from the last compartment of the desired strength for the soap-maker and can be used again for the saponification of the fats without the necessity of condensation and is of such purity that further treatment is not required.

The various anode-compartments containing the soap-lye or electrolyte solution are not connected necessarily. A pipe from the storage-vat containing the soap-lye runs directly above the electrolytical tank, with a spigot above each anode-compartment with a cock, by means of which the various anode-compartments are filled with the solution desired to be treated electrolytically. When a compartment becomes filled, the cock is closed and the liquor receives treatment until neutral, or until it has become sufficiently purified and clear, when an exit-cock at the bottom of the compartment is opened. This allows the treated solution, with precipitate and all, to run into a trough below, which trough conveys it to a tank, from where it is filtered in any way desired. The filtrate is now ready for the still. It is practically pure, is clear, of a slight yellow color, and being freed from the alkaline base and other impurities, glycerin can readily be distilled from it and in larger and purer quantities than if it had not been thus electrolytically treated.

The soap-lye or saponification liquor being a good conductor of electricity, from four to ten volts is amply sufficient to produce the electrolysis required; but in the electrolytical treatment of a crude-glycerin solution of a high density a higher electromotive force is required, according to the amount of active electrode-surface and the density of the glycerin solution.

A convenient and practical form of apparatus for carrying out our invention is illustrated in the accompanying drawings, in which—

Figure 1 is a plan view of the apparatus, and Fig. 2 is a longitudinal sectional view.

Similar letters refer to corresponding parts in both figures.

A is the diaphragms; B, the anodes; C, the cathodes; D, the main pipe which conveys the spent soap-lye solution; *d*, small pipe leading to the various anode-compartments; E, main electrical conductor for the anodes; F, main electrical conductor for the cathodes; G, exit-pipes for the treated solution; H, trough to convey treated solution for filtration; I, pipe to supply water to the first cathode-compartment; J, exit-pipe for the caustic solution; K, pipes connecting the bottom of the first cathode-compartment to the top of the next cathode-compartment, and so on throughout the length of the electrolytical vessel.

Having thus fully described our invention, what we claim as new, and desire to secure by Letters Patent in the United States, is—

1. The process of eliminating the caustic alkali from impure glycerin and spent soap-lyes or saponification liquors, and the decomposition of the glycerin compounds, which consists in subjecting the liquors to the action of a current of electricity, the anode being composed of zinc and being retained in contact with the liquor under treatment, substantially as described.

2. The process of extracting and recovering the caustic alkali from crude glycerin and spent soap-lyes or saponification liquors, which consists in subjecting the liquor to the

action of a current of electricity applied by means of an anode of zinc retained in contact with the liquor, and a cathode separated from the anode by a porous partition, substantially as described.

3. The process of eliminating the caustic alkali from impure glycerin and spent soap-lyes or saponification liquors, and the decomposition of the glycerin compounds, which consists in subjecting the liquors to the action of a constant current of electricity, the anode being composed of zinc and the cathode being composed of a metal such as lead or iron not attacked by caustic alkali, substantially as described.

4. The process of rendering spent soap-lyes or waste products from saponifications suitable for the distillation or production of glycerin, which consists in subjecting the impure glycerin solution to the action of a constant current of electricity, the current being applied by means of an anode of metallic zinc, filtering the precipitates formed by said action, and then distilling or condensing the filtrate, substantially as described.

5. The process of extracting and recovering the caustic alkali from crude glycerin and spent soap-lyes or saponification liquors, which consists in subjecting the liquor to the action of a current of electricity applied by means of an anode of zinc retained in contact with the liquor under treatment, and a cathode of a metal not attacked by caustic alkali immersed in water and separated from the liquor under treatment by a porous partition, substantially as described.

In testimony that we claim the foregoing as our invention we have signed our names in presence of two witnesses.

ORAZIO LUGO.
HUSON T. JACKSON.

Witnesses:
C. S. JACKSON,
CHAS. J. MARSH.