

# UNITED STATES PATENT OFFICE.

ALBERT DOMEIER AND OTTO CHRISTIAN HAGEMANN, OF LONDON, ENGLAND, ASSIGNORS, BY MESNE ASSIGNMENTS, TO JAMES S. KIRK & CO., OF CHICAGO, ILLINOIS.

## PROCESS OF RECOVERING CRUDE GLYCERINE FROM SPENT SOAP-LYE.

SPECIFICATION forming part of Letters Patent No. 475,758, dated May 31, 1892.

Application filed August 7, 1889. Serial No. 320,021. (No specimens.)

*To all whom it may concern:*

Be it known that we, ALBERT DOMEIER, merchant, and OTTO CHRISTIAN HAGEMANN, mechanical engineer, both of London, England, have invented a new and useful Improvement in Processes of Recovering Crude Glycerine from Spent Soap-Lye, of which the following is a specification.

The object of our invention is an improved process for recovering crude glycerine from spent soap-lye.

In carrying out our invention we take the spent soap-lye as it runs from the soap-coppers into a suitable tank and there add to and mix intimately with it a small proportion of caustic lime. The action of the lime on the lye is three-fold. First, it combines with soapy and fatty or resinous matters contained therein, forming insoluble lime-soaps; secondly, causticizes all or nearly all the carbonated alkali, thus bringing it into a proper form for removal by the means hereinafter described, and, thirdly, acts mechanically in weighting, and thus facilitating the precipitation of both the above-mentioned lime-soaps and any other flocculent or suspended matters originally present in the lye, the carbonate of lime resulting from the causticizing of the carbonated alkali also acting in this respect, as will be well understood. The quantity of lime to be used varies according to the quality of the spent soap-lyes under treatment; but we use enough to effect the aforementioned operations, the exact quantity being readily determined by the usual well-known chemical tests. We allow the precipitate thus formed to settle and draw off the clear liquor, filtering it, if necessary, into another suitable vessel—for instance, a soap-copper or the like—and next proceed to remove the excess of alkali from said liquor by adding to it an excess of tallow or fat or fat-oils or fatty acids and boiling the mixture until such fats or fatty acids have entered into combination with the excess of alkali in the liquor and removed it therefrom, or, by preference, we remove the alkali from the liquor by boiling it with an excess of rosin, as described in the patent granted to us on June 26, 1888, No. 385,105. In order that this removal of alkali may be the more fully understood, we will describe more in detail our procedure. In the case of the use of rosin

or a fatty acid—such as oleic acid—we preferably first concentrate the clear liquor until it reaches the point at which it is saturated with salts, or nearly so, and in this condition add to it the excess of such rosin or fatty acid, which will readily seize upon the free alkali, uniting therewith and forming soap, the large amount of salt held in solution by the liquor being sufficient to “salt out” the soap as fast as it is formed, while in the case of using neutral fats, which are not so readily saponifiable in the presence of a relatively large excess of salt in comparison to the alkali present in the liquor, we prefer to either boil the liquor in its natural state with such neutral fats or to concentrate it partially—that is, only to a less degree than in the case of employing the rosin or fatty acids—and thereby we secure a more complete removal of the alkali than would be the case if a much larger quantity of salt were present and which would result in the “opening” or “salting out” of the charge before the complete removal of the alkali from the liquor. At the end of the operation—i. e., when the fat has taken up the alkali from the liquor—in case the resulting soap is “close,” we, in order to separate and draw off the clear lye, boil the mixture “hard,” and thus evaporate some water from the liquor, causing the salt to accumulate in sufficient excess to salt out the soap, or we add at once to the charge the small excess of salt, (either in a solid form or in solution,) which may be necessary to effect the separation or salting out of the soap. We then remove the liquor to another tank or vessel for further treatment. If desirable, we may at this stage repeat the treatment of the liquor with a small quantity of lime and allow the resulting precipitate to settle and draw off or filter the clear liquor therefrom. This treatment is indicated when the liquor after the removal of the excess of alkali, as before described, is very frothy or opaque from the presence of soapy matters therein or contains much flocculent matter derived from the substances with which it has been boiled. We next add to the liquor a solution of alum or a chloride—such as the chlorides of iron, tin, or zinc—or a mixture of alum and a chloride or chlorides to effect the precipitation of the fatty and resinous acids in the liquor. We add such solution or solutions until no further



precipitate is produced. We allow this precipitate to settle and filter or draw off the clear liquor therefrom and next add to it a solution of caustic or carbonated alkali, which  
 5 has the effect of precipitating any excess of alum or chlorides which may have been used, together with any albuminous matters which may be present. These precipitates are allowed to settle and the clear liquor drawn off  
 10 or filtered therefrom ready for evaporation. In case an excess of alkali has been used in this last operation, it may be neutralized before or during the final evaporation by any convenient acid, such as hydrochloric or sulphuric  
 15 acid. We next convey the liquor to a suitable apparatus and concentrate by boiling until it has a specific gravity of about 1.300 (water equals 1.000) at 15° centigrade (or about 60° by Twaddell's hydrometer,) and thus produce  
 20 crude glycerine. During this operation we remove the salt which crystallizes out and wash it first with its own mother liquor and then with a solution of salt, by preference in the manner described in our application for  
 25 Letters Patent, Serial No. 294,307, dated December 21, 1888, (which Letters Patent were allowed on May 6, 1889,) thus rendering it of merchantable quality and fit for use again in various industries.

30 Having now fully described our invention, what we claim, and desire to secure by Letters Patent, is—

1. In the process of extracting crude glycerine and salt from spent soap-lye, the improvement which consists in first treating the spent  
 35 lye with lime and allowing the precipitate thus formed to settle and drawing off or filtering the clear liquor therefrom, then boiling the said liquor with fat, fatty acids, or  
 40 rosin to remove the free alkali therefrom, and then adding to the liquor alum or a suitable metallic chloride or a mixture of alum and a metallic chloride to precipitate fatty and resinous acids, substantially as described.

45 2. In the process of extracting crude glycerine and salt from spent soap-lye, the improvement which consists in first treating the spent lye with lime and allowing the precipitate thus formed to settle and drawing off or filtering the clear liquor therefrom, next reducing  
 50 the bulk by evaporation up to the point at which it is saturated with salt and boiling it with fat, fatty acids, or rosin to remove the free alkali, and then adding to the  
 55 liquor alum or a suitable metallic chloride or a mixture of alum and a metallic chloride to precipitate fatty and resinous acids, substantially as described.

60 3. In the process of extracting crude glycerine from spent soap-lye, the improvement which consists in first treating the lye with lime and allowing the precipitate thus formed to settle and drawing off or filtering the clear liquor therefrom, then boiling the said liquor  
 65 with fat, fatty acids, or rosin to remove the free alkali therefrom, and then adding to the liquor alum or a suitable metallic chloride or

a mixture of alum and a metallic chloride to precipitate fatty and resinous acids, removing the precipitate thus formed, and then adding  
 70 caustic or carbonated alkali to precipitate albuminous matters and any remaining alum or metallic chlorides, substantially as described.

4. In the process of extracting crude glycerine from spent soap-lye, the improvement which consists in first treating the lye with lime and allowing the precipitate to settle and drawing off or filtering the clear liquor therefrom, next reducing the bulk by evapo-  
 80 ration up to the point at which it is saturated with salt and boiling it with fat, fatty acid, or rosin to remove the free alkali and then adding to the liquor alum or a suitable metallic chloride or a mixture of alum and a metallic  
 85 chloride to precipitate fatty and resinous acids, removing the precipitate thus formed, and then adding caustic or carbonated alkali to precipitate albuminous matters and any remaining alum or metallic chlorides, substantially as set forth.

5. In the process of extracting crude glycerine from spent soap-lye, the improvement which consists in first treating the lye with lime and allowing the precipitate thus formed  
 95 to settle and drawing off or filtering the clear liquor therefrom, then boiling the said liquor with fat, fatty acid, or rosin to remove the free alkali therefrom and then adding to the liquor alum or a suitable metallic chloride or a mixture of alum and a metallic chloride to pre-  
 100 cipitate fatty and resinous acids, removing the precipitate thus formed, and then adding caustic or carbonated alkali to precipitate albuminous matters and any remaining alum or metallic chlorides, again removing the precipitate, and finally concentrating the clear liquor by evaporation to produce crude glycerine and salt, substantially as specified.

6. In the process of extracting crude glycerine and salt from spent soap-lye, the improvement which consists in first treating the lye with lime and allowing the precipitate to settle and drawing off or filtering the clear liquor therefrom, next reducing the bulk by evapo-  
 115 ration up to the point at which it is saturated with salt and boiling it with fat, fatty acid, or rosin to remove the free alkali, and then adding to the liquor alum or a suitable metallic chloride or a mixture of alum and a metallic chloride to precipitate fatty and resinous acids, removing the precipitate thus  
 120 formed, and then adding caustic or carbonated alkali to precipitate albuminous matters and any remaining alum or metallic chlorides, again removing the precipitate, and finally concentrating the clear liquor by evaporation to produce crude glycerine and salt, substantially as described.

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Witnesses:

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