## United States Patent Office,

JULIUS STOCKHAUSEN, OF CREFELD, GERMANY.

## PROCESS OF MAKING SOAP.

SPECIFICATION forming part of Letters Patent No. 646,326, dated March 27, 1900.

Application filed August 25, 1897. Serial No. 649, 429. (No specimens.)

To all whom it may concern:

Be it known that I, Julius Stockhausen, soap-manufacturer, of Crefeld, in the German Empire, have invented new and useful Improvements in the Manufacture of Soap for use in Dyeing and Finishing Textile Fabrics and Materials and for other Purposes, of which the following is a specification.

This invention relates to improvements in the process of manufacturing a soap of a gelatinous character from sulfonated fats, as described in the specification of Letters Patent

No. 585,347, dated June 29, 1897.

My present improvements consist, essentially, in heating to boiling temperature the sulfonated or sulfonized substance which, according to the data contained in the abovenamed specification of Letters Patent No. 585,347, has been mixed with a relatively-large amount of alkalies—that is, a quantity of alkali much larger than that used in the preparation of "Turkey-red oil." This quantity amounts to at least six per cent. of the sulfonized substance, while in the preparation of Turkey-red oil no more than two per cent. alkali is used.

Instead of fats oils, fatty acids, or oleic acids can be used. The alkalies can be replaced by other metal oxids, hydrates, or salts—as, for example, the oxids, hydrates, and salts of barium, strontium, magnesium,

and calcium.

I am aware that the saponification of fats by boiling with alkali is generally known and 35 that I already proposed in the second process described in the specification of the abovenamed Letters Patent to heat a mixture composed of a sulfonized oil or fat and an alkaline lye to 80° to 100° centigrade in order to 40 obtain a soap of gelatinous consistency. Still, it is unknown and perfectly novel to prepare a soap by boiling sulfonized fats, oils, fatty acids, or oleic acids in the presence of alkalies or the above-named other metal oxids, 45 hydrates, and salts. Now in order to bring the above-mentioned mixture to boiling it is necessary to submit it to a temperature of at least 102° centigrade, as the boiling-point is raised owing to the presence of the named 50 oxids, hydrates, or salts. Owing to this fact the temperature of 100° centigrade would in no way be sufficient, and it becomes necessary not only to expose the mixture to a higher temperature than 100° centigrade for | soap.

a short time, but to actually boil it, whereby 55 a heating to at least 102° centigrade is necessary. The thus-resulting product represents a considerable improvement with regard to the soap obtained according to the process described in my former patent. Through the 60 boiling operation named a more perfect saponifying of the sulfonized mass is obtained—that is to say, by boiling the various ingedients a product is obtained which is more perfectly saponified than the corre-65 sponding product which is prepared with the same amount of alkali without being boiled. Owing to this fact the resulting soap possesses still better qualities than the product obtained according to my older process.

The preparatory steps of the process can, for example, be as follows, as already described in the specification of my Letters Patent No. 585,347: One hundred parts, by weight, of castor-oil or of other similar or suitable 75 oil, oleic acid, fatty acids, or fats are mixed, with constant stirring, with about thirty parts, by weight, of sulfuric acid having a strength of about 66° Baumé. If the mixture is carefully made, no sulfurous acid should 80 be produced. The product is kept in a cool place for one or two days, with an occasional stirring. From whatever source the sulfonated substance may be obtained, it will hereinafter be called, for the sake of brevity, 85 "sulfonized oil."

In preparing the soap according to the present invention I may proceed in various ways, as will be indicated by the following examples:

Example I: To one hundred parts, by weight, of the sulfonized oil there are added sixty parts, by weight, of soda-lye of 36° to 37° Baumé all at once, with vigorous stirring. The mixture becomes clear when heat- 95 ed and has a yellowish color. It is allowed to stand for some days until the sodium sulfate (Glauber's salt) has crystallized out and the product from which the soap is to be obtained has precipitated. These two sub- 100 stances are then separated, and the second substance is boiled until it ceases to foam. For bringing the soap to boiling a temperature of no less than 102° centigrade is necessary, as already mentioned. A sample of the result- 105 ing product becomes of a gelatinous character when cold and exhibits the characteristics of

Example II: To one hundred parts, by ] weight, of the sulfonized oil there are added from one hundred to two hundred parts, by weight, of a lukewarm solution of common 5 salt of 25° to 30° Baumé for the purpose of separating the sulfuric acid and other substances. After vigorous stirring the mixture is allowed to stand for several days or as long as may be required until the oil has 10 been completely separated and can be removed by decantation. To one hundred parts of this decanted or separated oil there are added thirty-nine parts of soda-lye of 36° to 37° Baumé all at once with vigorous stir-15 ring. The mixture is then boiled by bringing it to a temperature of at least 102° centigrade, as described under Example I, until a cooled sample becomes of a gelatinous character and shows the characteristics of soap.

It will be evident that weaker solutions than those above specified may be employed in proportionately-larger quantities. In any case the product obtained is diluted soap of a gelatinous character, which may be comarcially used in this condition or be pre-

viously dried.

As already stated above, the new product shows to a still higher degree the useful qualities of the product obtained according to the process described in the specification of Letters Patent No. 585,347, because by boiling the various ingredients a product is obtained which is more perfectly saponified than the corresponding product which is prepared with the same amount of alkali without being boiled. Over all, the product made according to the present process possesses the quality of not being precipitated by salts,

and consequently upon the addition of large quantities of combination of earth alkalies, as it is specially necessary upon dyeing with substantive dyes, the same does not give rise to a precipitation and formation of a thin film of soap upon the surface of the water.

With the unboiled soap, however, a small amount of precipitation will always ensue, which is perfectly avoided with the boiled product. The unboiled product shows the same difference as compared with the boiled product in the process of sizing and finish-

product in the process of sizing and finishing upon addition of filling materials—as, for instance, sulfate of magnesia, sulfate of soda, and the like. While twenty parts of salt do not change solutions of the boiled product, twelve or thirteen parts are sufficient to cause turbidity with the unboiled

cient to cause turbidity with the unboiled one.

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Having thus described my invention, what I claim, and desire to secure by Letters Pat-

60 ent, is—

of a gelatinous character, consisting in sulfonizing fats, oils, fatty acids or oleic acid, treating the obtained product with a larger quantity of alkali than the one used in the preparation of Turkey-red oil, that is with a quantity of alkali amounting to at least six

per cent. of the sulfonized substance and heating the resulting product to boiling temperature, substantially as described.

2. The process for the production of a soap of a gelatinous character, consisting in sulfonizing fats, oils, fatty acids or oleic acid, treating the obtained product with a larger quantity of alkali than the one used in the 75 preparation of Turkey-red oil, that is with a quantity of alkali amounting to at least six per cent. of the sulfonized substance, allowing the sulfate of soda contained in the mass to crystallize out and heating the remaining substance to boiling temperature, substantially

as described.

3. The process for the production of a soap of a gelatinous character, consisting in sulfonizing fats, oils, fatty acids or oleic acid, 85 separating the sulfonated mass from the excess of sulfuric acid, treating the obtained product with a larger quantity of alkali than the one used in the preparation of Turkey-red oil, that is with a quantity of alkali amounting to at least six per cent. of the sulfonized substance, and heating the resulting product to boiling temperature, substantially as described.

4. The process for the production of a soap 95 of a gelatinous character, consisting in sulfonizing fats, oils, fatty or oleic acid, separating the sulfonated mass from the excess of sulfuric acid by means of common salt, treating the obtained product with a larger quantity of alkali than the one used in the preparation of Turkey-red oil, that is with a quantity of alkali amounting to at least six per cent. of the sulfonized substance and heating the resulting product to boiling temperature, 105

substantially as described.

of a gelatinous character, consisting in treating sulfonized fats, oils, fatty acids or oleic acid with a larger quantity of alkali than the 110 one used in the preparation of Turkey-red oil, that is with a quantity of alkali amounting to at least six per cent. of the sulfonized substance and heating the obtained product to boiling temperature, substantially as de-115 scribed.

6. The process for the production of a soap of a gelatinous character consisting in treating sulfonized fats, oils, fatty acids or oleic acid with a larger quantity of alkali than the one used in the preparation of Turkey-red oil, that is with a quantity of alkali amounting to at least six per cent. of the sulfonized substance, allowing the sulfate of soda to crystallize out and heating the remaining 125 product to boiling temperature, substantially as described.

In witness whereof I hereunto set my hand in presence of two witnesses.

## JULIUS STOCKHAUSEN.

Witnesses:

GEORGE GOMPERTZ, ERNEST JULIUS SCHREIBER.