

UNITED STATES PATENT OFFICE.

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PROCESS OF MAKING SOAP.

SPECIFICATION forming part of Letters Patent No. 585,347, dated June 29, 1897.

Application filed February 26, 1896. Serial No. 580,507. (No specimens.)

To all whom it may concern:

Be it known that I, JULIUS STOCKHAUSEN, a subject of the King of Prussia, Emperor of Germany, residing at Crefeld, in the Kingdom of Prussia and German Empire, have invented certain new and useful Improvements in Processes of Making Soap, of which the following is a specification.

The gelatinous soap made according to my improved process now to be described presents the following great technical advantages which are of special interest to the textile industries and dyeing:

The soap can be used to its greatest advantages in the hardest waters without requiring a previous softening of the water. By adding a comparatively small quantity of the soap the bath becomes as clear as crystal in a few minutes when warmed.

If the soap-baths used in dyeing have become decomposed, through any circumstances whatever, they are revived to full usefulness by the addition of a small quantity of the gelatinous soap produced by my process. Even such baths which show separations of fatty acids produced by common soap are rendered entirely useful by the addition of a small quantity of the new soap produced by the new process, whereas a dyeing-bath prepared solely with the new soap remains clear from beginning to end.

The use of soap-bark soap heretofore required in silk-dyeing will no longer be necessary if the gelatinous soap of my process is used. Such methods of dyeing in which the soap-bark soap was essential on account of its acid reaction produce much better results with the gelatinous soap and without the soap-bark soap, and the baths never show any injurious decomposition, which is of still greater importance. Baths prepared with the soap in question further have the property of fixing the coloring-matter added to the bath clearly and intensely on the fabrics and yarns. For coloring silks black the soap-bath produced by means of the gelatinous soap is of a value heretofore not attained.

The defect which always appears and removal of which has always been unsuccessfully attempted, that the fresh appearance of black and other colors has been marred by turbidities from a slight shade to crystalline

appearance, cannot occur if soap produced by my improved process is used.

The elasticity and strength of the yarns and fabrics of all kinds, above all silk, are not injuriously affected in the least by the use of this soap, but on the contrary a rich body of the thread—that is, a swelling of the same—is attained with an uncommon flexibility. All these technical advantages are essentially attained by sulfurizing fats, oils, fatty acids, and oleic acids, the surplus of sulfuric acid being separated and the mass obtained being saponified, so that a gelatinous soap of acid reaction is obtained. This soap may be used for all purposes, but presents special advantages for technical uses, and among these for the purposes of dyeing, bleaching, and printing.

The separation of the sulfuric acid is accomplished best by neutralization and crystallization of the sulfates or by converting the sulfurized fats or oils into salts. According to the requirements of the industry this acid soap can be converted into a neutral or basic soap by a corresponding addition of alkalis.

In practice my improved process is carried out as follows: One hundred parts, by weight, of castor-oil (or oleic acid, fluid oils, or solid fats) are separated into four to six lots, and in intervals of from one to four hours are mixed with thirty parts, by weight, of sulfuric acid of 66° Baumé under constant stirring, but care must be taken to prevent the formation of sulfurous acid. This mixture is permitted to remain in a cool place for one to two days and during this time is stirred several times. The sulfurization of the oil or fat can also be accomplished by means of sulfonic acids. Out of this preparation the new gelatinous soap is made in the following manner:

First process. To one hundred parts of this preparation sixty parts of caustic soda-lye of 36° to 37° Baumé, as free as possible of carbonic acid, are added in one lot either hot or cold, and the mixture is thoroughly and constantly stirred. This mixture is permitted to stand for from four to eight days until crystallization is completed or the completed soap has separated from the crystals. An acid soap is thus obtained which heretofore has not been possible in the industries. To neutralize this acid soap according to the

requirements of the respective industries, a further quantity of the above-mentioned caustic soda-lye of 36° to 37° is added and further addition of the lye produces an alkaline soap. In this manner acid, neutral, or alkaline soaps can be made by this process. The soap produced consists of a gelatinous mass. If for any reason it is desired to have the soap in a more liquid state, this can easily be accomplished by adding water.

Second process. To one hundred parts of the above-mentioned preparation one hundred to two hundred parts of a lukewarm solution of common salt of 25° to 30° Baumé are added for the purpose of eliminating any surplus free sulfuric acid or dirt or impurities. The mixture is stirred until the salt water has absorbed the above-named impurities and then the mixture is permitted to stand for one or two days, if necessary. Then the salt-lye and impurities are drawn off. To each one hundred parts of the cleaned preparation thirty-nine parts of caustic soda-lye of 36° to 37° Baumé, as free as possible of carbonic acid, are added in one lot. The mixture is then heated to 80° to 100° centigrade for obtaining a better chemical combination. The soap obtained is also acid and of gelatinous consistency. This soap can also be converted into a neutral or alkaline soap by the addition of lye, and it can be thinned by the addition of water.

The soaps obtained by the first and second processes can be mixed with each other or with suitable quantities of other neutral or alkaline soaps, according to the different branches of industry. The saponification of the above-mentioned preparation or other sulfonated oils, fats, or oleic acids can be accomplished by means of caustic potash, caustic ammonia, carbonate of soda, carbonate of potash, carbonate of ammonia, alkaline earths, metal oxides, hydrates, or carbonates.

The described process may be also used for softening hard water or for the purpose of preventing the formation of boiler-scale.

The proportions of ingredients above given are by weight.

I am aware that it has been proposed to alter, by treatment with sulfuric acids, the glycerin contained in the oils and fats into a substance which combines with alkali and thus enters into the process of saponification. In the common process of saponification the glycerin produced out of the oils and fats by the saponification is separated, namely, up to five per cent. of glycerin, according to the nature of the fats. In olden times, when the value of glycerin was not yet known, this glycerin was judged to be offal or loss. By the addition of six parts of sulfuric acid of 66° Baumé to one thousand parts of oil or fat, as heretofore proposed, the glycerin contained in the oil would be transformed into sulfo-

glycerin acid. In my process, however, I transform all the oil or fat, by an addition of thirty parts sulfuric acid at 66° Baumé to one hundred parts of oil or fat, into a homogeneous completely-sulfonated mass, and it is not my intention to saponify the glycerin. The object of my invention is, on the contrary, to create by the saponification of a perfectly-sulfonized oil a soap of an acid reaction and a gelatinous consistency which will be of great importance to the whole textile industry. Heretofore the glycerin has been preserved in the soap by preventing it from entering the lye, with no other purpose than to increase the weight, and by the presence of this glycerin the action of the soap is not at all altered, nor is the soap improved; but this old method does not pay now that the glycerin has obtained a great importance, (*e. g.*, in the manufacture of nitroglycerin, &c.) To-day big companies have grown up for making the costly glycerin by means of disassociating all sorts of fats and oils, and only afterward the oleic acids produced by the disassociation of fats and oils are saponified.

In all important soap-houses where no previous disassociation of fats takes place the glycerin coming out after saponification is obtained by evaporating the remaining lye. For this purpose great chemical works have been established to obtain the glycerin from the remaining lye. Therefore in no soap-houses to-day is glycerin saponified, but everywhere the glycerin is recuperated.

Having thus described my invention, what I claim, and desire to secure by Letters Patent, is—

1. The process for making soap of an acid reaction and a gelatinous consistency, which consists in completely sulfonizing fats, oils, fatty acids or oleic acids, separating the obtained product from the remaining sulfuric acid, and perfectly saponifying the sulfonized mass by a quantity of alkali equivalent to the existing fatty acids, freeing the produced soap from fatty acids and alkali, as set forth.

2. The process for making soap of a gelatinous consistency, which consists in completely sulfonizing fats, oils, fatty acids or oleic acids, separating the obtained product from the remaining sulfuric acid, perfectly saponifying the sulfonized mass by a quantity of alkali equivalent to the existing fatty acids, and finally transforming the acid soap produced into neutral or alkaline soap by a corresponding addition of alkali, as set forth.

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

JULIUS STOCKHAUSEN.

Witnesses:

TH. WALDAPFEL,
WILLIAM H. MADDEN.