UNITED STATES PATENT OFFICE.

ERNST TWITCHELL, OF WYOMING, OHIO.

SULFO-FATTY-AROMATIC ACID SALT.

1,170,468. Specification of Letters Patent. Patented Feb. 1, 1916.

No Drawing. Application filed January 29, 1913. Serial No. 744,820.

To all whom it may concern:

Be it known that I, ERNST TWITCHELL, a citizen of the United States, residing at Wyoming, in the county of Hamilton and State of Ohio, have invented certain new and useful Improvements in Sulfo-Fatty-Aromatic Acid Salt, of which the following is a specification.

My invention relates to a new product comprising a salt of a saponifying (hydrolyzing) acid reagent adapted to be used for the treatment of fats and oils in the manufacture of fatty acids and glycerin.

The object of the invention is to provide a commercial saponifier in the shape of a dry, stable powder, constituting a sulfonic material having a sulfonic acid in combination, which can be boiled with fats or oils in treatments for the separation of glycerin and fatty acids, in the presence of a mineral acid which will react on the reagent to liberate an active, stable, pure and concentrated, sulfonic acid saponifier, of an oily nature, and which will not decompose at the temperature of boiling water under which the process is carried on, and the metal component of the reagent uniting with the acid to form a product capable of being separated from the glycerin as a precipitate. This sulfonic material, being a stable powder or solid, can be safely shipped in ordinary packages and does not require the use of sealed vessels, as when the conventional liquid saponifier is used. The dry solid sulfonic material can be more efficiently administered in the treatment than a liquid saponifier, and can be used in smaller quantities. But of still greater importance is the fact that the sulfonic acid when so liberated from the combination existing as a dry solid or powder in the presence of the boiling fats or oils, becomes a concentrated, pure and stable saponifier, far more intense in its saponifying action than any liquid material adapted and used for the same purpose.

More specifically, it is the object of my invention to refine and concentrate the crude product known generically as sulfo-fatty-aromatic acid, such a product and a process of producing it being described and claimed in my prior Patent No. 682,503, dated July 11, 1899.

A specific example of this saponifying reagent may be designated as naphthalene-stearo-sulfonic acid, and this example will serve to illustrate the principles on which my present invention is based, the conversion of said product into a salt constituting one of the concrete embodiments of the series of products which it is the object of this invention to produce. Under manufacturing conditions obtaining, this product when produced in accordance with the process described in my said prior patent, exists as about a forty per cent. component of a viscous, dark-colored liquid, containing as impurities approximately about thirty-five per cent. of oleic acid, which has been acted upon, polymerized and colored by the sulfuric acid, and approximately twenty-five per cent. of water containing some sulfuric acid and naphthalene-sulfonic acid. This crude product is an oily or tarry liquid almost black, and while it is and has been for many years advantageously employed in the manufacture of fatty acids and glycerin, it is open to the objection that the active saponifying acid exists in a non-refined and non-concentrated condition. Also the oil slightly colors the fatty acid in the ultimate saponifying process, while the naphthalene-sulfonic acid somewhat contaminates the glycerin. Furthermore, inasmuch as this crude product exists as a heavy liquid, it requires shipping in sealed vessels the bulk of the product being considerable in proportion to the weight of the active saponifying acid component.

It is the object of my present invention to refine and concentrate this crude product and convert it into a water-insoluble salt of the naphthalene-stearo-sulfonic acid, which can be separated, dried and powdered and shipped in condensed form. The preferred process of treating this crude product constitutes the subject-matter of a separate application for Letters Patent, filed January 29, 1913, Serial No. 744,818. It will suffice for the present purpose to briefly recite the steps of this process as it is preferably employed.

The crude product of the naphthalene-stearo-sulfonic acid is first treated with a solution of common salt in order to wash out the greater part of the sulfuric acid and aromatic sulfonic acid which it contains as impurities. This treatment also converts part of the sulfo-fatty-aromatic acid into its sodium salt (water alone would not do for this washing as the reagent is soluble in

pure water, but insoluble in water containing salt). Second, the material is put into a suitable extraction apparatus and extracted with naphtha or other solvent which dissolves fats and fatty acids but not the sulfo-fatty-aromatic acid. This operation removes the free fatty matter which had been acted on and colored by the sulfuric acid in the manufacture of the original reagent but had not been converted into sulfo-fatty-aromatic acid. Operations one and two may be advantageously combined into one. Third, the extracted material is dissolved in a large quantity of water and precipitated with a salt of such a metal as will form an insoluble compound with the fatty-aromatic-sulfonic acid—such metals are barium, calcium, aluminum, magnesium, etc. As the sulfo-fatty-aromatic acid has not been completely neutralized with the salt water treatment, it may be necessary at this stage to add a small quantity of a base to neutralize the mixture and cause complete precipitation. It is to be understood, however, that the neutralizing step need only be sufficient to neutralize the sulfonic component of the crude product, as the fatty acid component, being a relatively weak acid, need not be completely neutralized. The remainder of the soluble impurities have been washed out by the process. Fourth, the precipitate, which sinks to the bottom of the tank as a viscous mass, is removed, dried and ground to powder.

The powder is stable and the major portion comprises a concentrated saponifying agent. To test the strength of this product it is only necessary to determine the percentage of the sulfonic component thereof (radical, SO_3H) as this constitutes the active saponifying principle of the material.

The residue of my new product, amounting to approximately fifteen or twenty per cent., consists of oil or a little water, but without any appreciable deleterious characteristics.

I thus obtain a solid which can be powdered as distinguished from the liquid product. The powder is non-hygroscopic, can be cheaply packed for shipping without danger of loss through leakage, and it is extremely stable.

I thus obtain a condensed, concentrated and stable sulfonic material, existing as a commercial dry solid or powder, the active, sulfonic saponifier of which is relatively double strength as compared to the original or crude product, and it can be administered as a powder or broken up solid in the treatment process, in connection with a sufficient amount of mineral acid, such as sulfuric or hydrochloric acid, to react upon the material and liberate the active, stable sulfonic acid saponifier, the metal component of the material forming a combination with the mineral acid which can be separated out as a precipitate. This material can be stored indefinitely, shipped conveniently in reduced bulk as a dry solid, and used conveniently and economically when required, the saponifier efficiency is increased, and a better product produced. This powdered form of reagent is of a dull-grayish color, as a considerable amount of the dark-colored oil has been eliminated, so that lighter products result from the use of this new reagent, and as all of the naphthalene-sulfonic acid has been removed, the glycerin produced as a result of the use of my present reagent is much purer than that obtained by using the crude reagent.

Having described my invention, I claim:—

A sulfonic material for the manufacture of glycerin and fatty acids from fats and oils, consisting of a dry, stable solid, insoluble in water, consisting of a neutral body having the property when treated with a mineral acid of yielding an active, concentrated sulfonic saponifier, of an oily nature, which will not decompose at the temperature of boiling water, and a residue of innocuous character adapted to be removed as a precipitate.

In testimony whereof, I have hereunto set my hand.

ERNST TWITCHELL.

Witnesses:

OLIVER B. KAISER,

LOUISE A. BECK.