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SULFURIZED FATTY AND WAXY MATERIALS

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> (CI. 260-399) 1 Claim.

The present invention relates to sulfurized fatty and waxy materials.

Although not limited thereto, the present invention deals with the sulfurizing of lanolin, wool, grease or "degras" that has been partially or completely saponified and consists of a mixture of salts and uncaponifiable matter.

It is among the objects of the present invention to provide a simple, inexpensive process for sulfurizing fatty and waxy materials and particu- 10 larly wool grease or "degras" to give increased yields of highly sulfurized products with less conversion to undesirable by-products.

Another object is to provide a novel sulfurized wool grease or "degras" which may be most ef- 15 fectively utilized as an additive for mineral oils so that they may be used in high pressure lubricants and also generally in motor oils and other lubricating combinations.

Still further objects and advantages will ap- 20 pear in the more detailed description set forth below, it being understood, however, that this more detailed description is given by way of illustration and explanation only and not by way of limitation, since various changes therein may be 25 made by those skilled in the art without departing from the scope and spirit of the present invention.

According to the present invention, it has been found most satisfactory first to heat the lanolin, wool grease or "degras" in the presence of a strong alkali partially or completely to saponify the same. This partially or completely saponified material is then mixed with a mineral oil with or without previous drying and after removal of the water, is treated with a sulfurizing agent, a phosphorus sulfide being preferred.

If desired, as a last step, a heavy metal salt may be added in the form of a water soluble salt and a sulfurized, metallized wool grease may thus be produced.

To give a specific example of the above procedure:

Example I

A wool grease or "degras" is selected, contain- 45 ing up to 15% free fatty acids, 5 to 10% free fatty alcohols and 75 to 95% fatty acid esters.

This wool grease or "degras" is then heated to a temperature of about 270° to 280° F. in the presence of about 10% caustic soda for a period 50 of about one hour in the presence of a minimum amount of water.

Generally, 100 parts by weight of wool grease may be first heated to a temperature of 210° F. Then 10 parts by weight of caustic soda are added 55 of ways. For example, the product may be

in three portions, over a period of fifteen minutes with stirring, the caustic soda being dissolved in a minimum quantity of water. The temperature during this operation is gradually raised about 1° F. per minute to a final temperature of 275° F. At about the end of one hour the major portion of the water is driven off.

The hot reaction mixture will consist of the sodium salts of the wool grease acids, unsaponifiable matter, some unchanged esters, and a small amount of water.

The hot reaction mixture may be dried in at about 105° C. or more rapidly under vacuum. If desired, however, the product may be dried by first adding it to a lubricating oil and blowing with air at an elevated temperature.

For example, between 100 and 125 parts by weight of the product may be placed in about 75 parts by weight of mineral oil and the temperature raised gradually up to 275 to 300° F.

As the temperature is raised, air is blown through until substantially all the moisture has been removed. The temperature is then regulated to about 285° F.

Then a slurry is prepared of about 23 parts by weight of phosphorus pentasulfide in 25 parts of mineral oil. About 12 parts of the slurry are added with stirring to the mixture, while the mixture is held at a temperature of 250° F. to 350° F., but preferably at 285 to 300° F.

Some foaming will result which will increase as additional amounts of the slurry and phosphorus pentasulfide are added.

The remainder of the slurry may be added, about 12 to 13 parts by weight at a time, at intervals of five minutes, and the temperature is maintained at 285 to 300° F. for about one hour, during which the foaming will subside.

After heating for about one hour, the reaction mixture is cooled and filtered to give a relatively high yield of sulfurized degras of about 215 parts by weight as compared with the original amounts of wool grease utilized, which was about 100 parts by weight.

It is often desirable to add heavy metal salts, such as lead, and this may be done by combining about 100 parts by weight of sulfurized alkali treated wool grease with about 24 parts of lead acetate. Specifically, 100 parts by weight of sulfurized alkali treated wool grease or "degras" are heated to approximately 200° F. A solution of 24 parts by weight of lead acetate in about 24 grams of water is added to the mixture, heated to about 200 to 210° F. for 45 minutes.

The acetic acid may be removed in a number

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cooled and excess water added, followed by heating in a steam bath and stirring. After cooling, the wash water may be decanted and after three or four washings, the product may be dried at 212° F.

The residual acetic acid and water may also be removed by raising the temperature of the product to 210 to 220° F. followed by blowing with air for 45 minutes. Generally, the blowing-air process is preferred as a method of removing 10 water and acetic acid.

The final composition will be a most desirable additive to motor oils and to high pressure lubricants, such as those used on gears.

possible to utilize compounds of the following metals and particularly their water-soluble salts for metallizing purposes, these metals being:

Tin Mercury Silver Iron Nickel Copper Cadmium Lead

Instead of or in mixture with the wool grease or "degras," it is also possible to utilize the following fatty or waxy materials, particularly of ester form such as vegetable, animal and fish oils and waves, namely:

Waste grease Carnauba wax Cottonseed oil Japan wax Peanut oil Sperm oil Whale oil Coconut oil Spermaceti Lard oil Soya bean oil Tallow

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In combination with the sulfurized degras, it is also possible to utilize various alkyl and aryl thiophosphates or thiophosphites. Instead of phosphorous pentasulfide in addition thereto it

is also possible to use phosphorous sesquisulfide.

The embodiment of the invention shown and described herein is to be considered merely as illustrative, as the invention is susceptible to variations, modification and change within the spirit and scope of the appended claim.

Having now particularly described and ascertained the nature of the invention, and in what manner the same is to be performed, what is claimed is:

A process of making leaded sulfurized saponified degras which comprises heating wool grease to about 270° to 280° F. in the presence of about 10% caustic soda for about one hour, and the In lieu of or in addition to lead salts, it is also 15 caustic soda being in a concentrated solution in a minimum amount of water, drying the hot reaction mixture by adding it to a lubricant oil and blowing with hot air at a temperature of about 285° F., about 100 to 125 parts of the reac-20 tion mixture being placed in about 75 parts of the mineral oil, then mixing with a slurry of about 23 parts of phosphorous pentasulfide and 25 parts of mineral oil and maintaining with stirring a temperature of 285° to 300° F. and then treating the thus sulfurized saponified wool grease with a solution of lead acetate at a temperature of about 200 to 212° F. and finally removing the acetic acid

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