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IMPROVED MANUFACTURE

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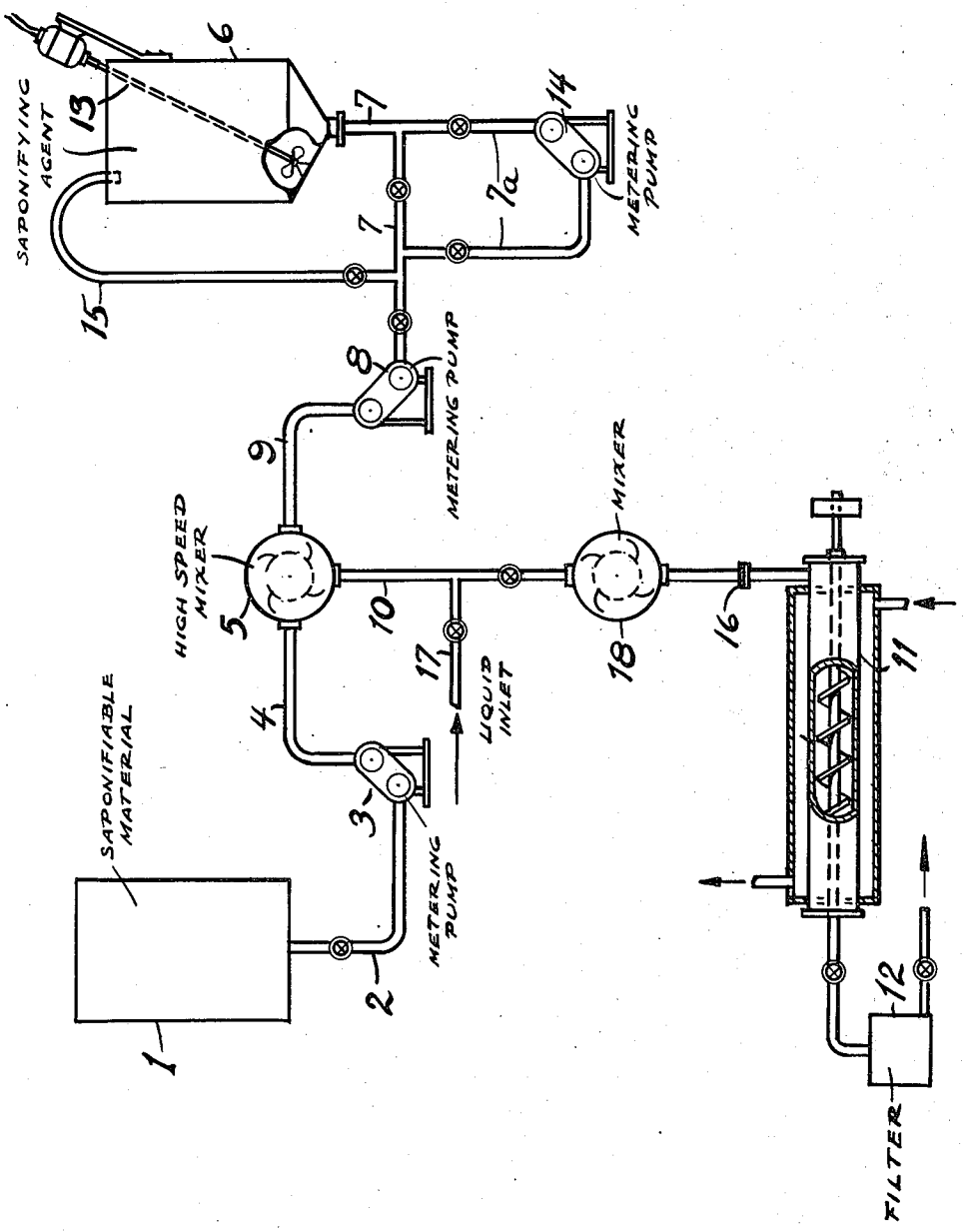

John C. Zimmer Inventors
Arnold J. Morway
By P. C. Young Attorney

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IMPROVED MANUFACTURE

John C. Zimmer, Union, and Arnold J. Morway, Clark Township, Union County, N. J., assignors to Standard Oil Development Company, a corporation of Delaware

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11 Claims. (Cl. 252—39)

This invention relates to the novel method of manufacturing soap-thickened compositions. More particularly it relates to a continuous process for reacting a saponifiable material with a spanonifying agent in the presence of a liquid medium.

There have been many difficulties involved in the preparation of various soap-thickened organic liquid compositions because the thickening effect of the soap tends to increase the viscosity rapidly in the immediate vicinity of the reacting particles and tends to produce results which are not uniform. This is partly due to the gel structure of the soap in the presence of the organic liquid medium and partly due to the fact that the soap constituents are not entirely soluble in the organic liquids.

It has now been found that if the saponifiable material and the saponifying agent, in the presence of the organic liquid medium, are mixed together with sufficient speed and efficiency that the process can be carried out continuously with the production of a uniform product of high quality. Thus it is one object of the invention to manufacture soap-thickened compositions continuously with great speed and efficiency. Another object of the invention is to carry out this continuous manufacture with a minimum lay-out of plant equipment and with a relatively simple, fool-proof design so that the plant can be operated for maximum production with a relatively small amount of labor, and with a relatively low requirement of skilled labor and highly technical personnel.

Broadly, the invention comprises continuously mixing together proportioned amounts of a liquid medium containing a saponifying agent dispersed therein and effecting this mixing in a high speed mixer in which said two liquids are contacted with each other in a highly turbulent zone, whereby efficient dispersion thereof is obtained by means of the shearing action of rapidly moving parts.

For example, in applying the invention to the manufacture of a lubricating grease, the saponifiable material, such as a fatty acid or fat, is dissolved in a portion of mineral lubricating oil, and hydrated lime or other suitable saponifying agent is suspended in another portion of mineral lubricating oil and then these two liquids, preferably each preheated to the desired saponification temperature, are fed through suitable proportioning equipment, such as metering pumps, into a high speed mixer, such as a Lancaster disperser, where the saponifiable reaction is completed almost immediately due to the extreme speed and efficiency of the mixing.

For the preparation of lubricating greases, the liquid medium in which the saponifiable material and the saponifying agent are separately dissolved or dispersed, should be a hydrocarbon liquid having various physical properties, depending upon the intended use of the grease, such as:

Flash point of 200° to 600° F.
Viscosity, about 35 to 250 seconds Saybolt Universal at 210° F.
V. I., about 5 to 120.

Such mineral oil may be derived from various types of crudes, such as paraffinic, naphthenic, asphaltic, etc., and may be subjected to any desired type of refining, such as distillation, clay treating, acid treating, solvent extraction, etc.

The saponifiable material to be used may be any of the natural or synthetic fats, fatty acids, fatty oils, etc., suitable for use in making lubricating grease. Specific examples of such materials include beef stearin, mutton tallow, hog fat, oleic acid, degras, stearic acid, tallow fatty acids, degras fatty acids, lard oil, rosin, soya bean oil, cottonseed oil, corn oil, neat's-foot oil, and similar animal and vegetable oils or various hydrogenated fatty oils, such as hydrogenated fish oils, or fatty acids, derived from such fats and oils as cottonseed oil acid, hydrogenated fish oil acids, etc., or synthetic acids, such as those produced by the oxidation of paraffin wax. Other grease forming carboxylic acids may be used such as the naphthenic acids derived from petroleum oils, especially those boiling in the range of kerosene, gas oil, or higher fractions.

The saponifying agent may be lime, either in the form of calcium hydroxide, which is preferred, or calcium oxide, or other alkaline earth metal bases, such as barium hydroxide or strontium hydroxide or magnesium hydroxide or -oxide, aluminum hydroxide, lead oxide as well as alkalies, such as lithium, sodium, potassium, rubidium, and cesium, and other known saponifying agents.

The saponifying material and the saponifying agent should normally be used in neutralizing proportions although for some purposes, it is desirable to have a slight excess of acid and for others a slight excess of the saponifying agent. The proportions of oil and soap in the finished grease will, of course, depend upon the intended use as well as the types of soap and the viscosity and other characteristics of the lubricating oil, but in general the soap should constitute about

5% to 50%, and preferably about 10% to 30%, by weight of the finished grease.

One particular type of lubricating grease composition adapted to be made according to this invention, is the one which is claimed in our copending application Serial No. 415,870, filed October 21, 1941, now Patent #2,332,825, which is intended especially for lubricating anti-friction bearings, automotive or tractor chassis, air-brake equipment, etc. The lubricant for the latter use must have a pour point at least as low as —25° F. and must have a low temperature co-efficient, i. e. a low viscosity change with temperature, or, in other words, should have a high viscosity index and should be semi-fluid at all times; furthermore, the oil used must not swell rubber, because the grease comes in contact with rubber bushings and gaskets, and the soap should not separate from the oil. The particuar lubricant is preferably composed of about 20% or so of a lime soap of hydrogenated fish oil acids, and about 60% to 80% of a low pour point aromatic-free coastal lubricating oil, preferably together with a small amount such as 0.5% to 20% of a soluble organic thickener such as a linear olefinic polymer, e. g. polyisobutylene having a molecular weight of about 5,000 to 25,000, preferably about 10,000 to 15,000. The hydrogenated fish oil fatty acids are superior to stearic acid for this particular purpose because they contain a substantial proportion of 20 or 22 carbon atoms per molecule as compared to commercial stearic acid which only contains 18 and 16 carbon atom acids. Various grades of hydrogenated fish oil acids are available on the market under various trade names, such as Hydrofol, Wyandotte, Weckostear, Hydres, Snowdotte. The preferred type is a hydrogenated fish oil acid having the following pertinent specifications: titre 52° C., free fatty acid (as oleic) 99%, iodine value 6, saponification No. 194 (Hydrofol 51).

It is, of course, understood that the lubricating greases made may contain a small amount of water or glycerine, either of which may be present as a result of the saponification reaction or may be added, if desired.

The present application is a continuation-in-part of said earlier application, Serial No. 415,870, filed October 21, 1941, now Patent #2,332,825.

The invention also has many other important applications, such as the production of soap-thickened pastes, solid fuel compositions. These may be made, for instance, by thickening an inflammable hydrocarbon oil, such as gasoline and/or kerosene, or even heavier fractions, such as gas oil and lubricating oil, with a suitable object which may be sodium stearate, sodium abietate, sodium or calcium soap of hydrogenated fish oils, lithium soaps of stearic acid or hydrogenated fatty oils or acids or the hydrogenated soaps of abietic acid.

In the various soap-thickened compositions which can be prepared according to this invention, various optional addition agents may be used, such as the oil-soluble thickeners of the substantially linear polymer type, e. g., polyisobutylene, polymethacrylate esters, polyvinyl butyl ether, etc., which should have a relatively high molecular weight, e. g., 1,000 to 100,000, or more. Also organic swelling agents may be used, such as rubber or synthetic substitutes therefor, although, of course, such materials which are highly unsaturated could only be used where this high unsaturation, which tends to make the products susceptible to oxidation and gumming, would not be objectionable. Other optional addition agents include amine, phenolic or amino phenolic oxidation inhibitors, degras, degras alcohol, saturated or unsaturated alcohols of more than 3 carbon atoms, glycols, polyglycol, glycol ethers, etc., dyes, pigments, extreme pressure lubricating agents, amine or Schiff's base metal deactivators, etc.

For some purposes, it is satisfactory to use a liquid medium which is not simply a hydrocarbon liquid but contains other chemical elements, such as, oxygen, halogen, etc., as, for instance, di-butyl phthalate, chlorinated di-phenyl (available on the market under the trade-name "arochlor"), chlorinated benzene, chlorinated alcohol, chlorinated kerosene, chlorinated paraffin wax, ethers, ketones, glycerines, etc.

The procedure to be used in carrying out the present invention may be varied, to some extent, depending upon whether all of the liquid medium, such as the lubricating oil or gasoline, etc., is present during the saponification reaction or only part of which is present then and the rest is continuously blended in later; for satisfactory blending in such latter case, it is preferable that at least 20%, and preferably at least 30% or more, of the liquid medium be present during the saponification.

Of the total amount of lubricating oil or other liquid medium present during the saponification, generally about 50 to 90%, preferably 70 to 80% thereof, should be used as the solvent or dispersing agent for the saponifiable material, and the remainder as the dispersing medium for the saponifying agent. The governing factor in determining how much of the oil or other liquid medium should be used in each case, is the desirability of having each of the two liquids of suitable fluidity or consistency for rapid and continuous, i. e. uninterrupted, flow from the supply source through the pipe lines, proportioning equipment, and final mixer. There should be enough oil with the saponifiable material to completely dissolve it and make a resulting solution which will flow satisfactorily at the desired saponification temperature; generally for every one part by weight of saponifiable material, about 5 to 9 parts by weight of lubricating oil, gasoline or other suitable liquid medium should be used and similarly for every part by weight of saponifying agent, about 3 to 5 parts by weight of lubricating oil, gasoline or other suitable liquid medium should be used.

The temperatures to be used will, of course, depend primarily upon the particular materials used as well as to some extent upon other factors such as the pressure, etc. For instance, in the preparation of lubricating greases generally a temperature of about 120° to 150° F. is desirable for dissolving the fatty acid or fat in the lubricating oil base stock and the temperature used during the saponification will depend primarily upon the type of saponifying agent used as, for example, for making a lime soap grease the temperature should be from about 120° to 150° F., whereas for a soda soap grease, the temperature should be about 300° to 500° F. For best carrying out the present invention, the dispersion of saponifying agent in lubricating oil should also be heated to the saponification temperature and the solution of saponifiable material in oil should be either heated or cooled as necessary to bring it to approximately saponification temperature. If desired, one of these two liquids may be preheated to a temperature sufficiently above the

saponification temperature to compensate for the use of the other liquid at a correspondingly lower temperature.

Obviously when soap-thickened compositions are prepared from more volatile hydrocarbon liquids, such as gasoline, or other organic liquids, it may be desirable to carry out the saponification under pressure to avoid undue volatilization.

The saponification will generally be carried out under atmospheric or under slightly super-atmospheric pressure, as for instance, up to 15 lbs./sq. in., or so, especially in the case of manufacturing lime soap grease where it is desirable to retain in the grease some of the water formed by the saponification reaction in order to obtain the desired grease structure. For preparing soap-thickened compositions using gasoline as the liquid medium or as the chief constituent thereof, a super-atmospheric pressure of about 20 lbs. to 50 lbs./sq. in., or higher or so should be used with a corresponding temperature of about 120° to 160° F. when using lime as the saponifying agent. Sub-atmospheric pressure may be used if it is desired to withdraw water or other by-products formed during the reaction.

In order to illustrate the operation of the invention, reference is made to the accompanying drawing which is a schematic lay-out of suitable equipment for carrying out the invention.

Referring to the drawing, a solution of saponifiable material in a suitable liquid medium is continuously withdrawn from supply tank 1 through line 2 and metering pump 3 via line 4 into the high speed mixer 5 where it is rapidly and intimately mixed with a dispersion of saponifying agent in a suitable liquid medium, which mixture is continuously withdrawn from supply tank 6 through line 7, metering pump 8, and line 9. The saponification reaction takes place within the high speed mixer 5 and the resultant soap-thickened composition issues continuously through line 10 and may, if desired, be passed through a cooler 11, which is preferably a scraped chiller of the Carbondale type, and preferably the product is also passed through a filter 12 from whence it is discharged continuously into suitable containers (not shown) for shipment or storage. The supply tank 6 is preferably provided with a suitable agitator 13 (driven by a suitable source of power not shown) in order to prevent settling of dispersed saponifying agent and in order to maintain the dispersion uniform. Also, if it is desired to still further insure uniformity in the dispersion of saponifying agent in the liquid medium in supply tank 6, one may provide a recycling pump 14 in line 7 and a line 15 for leading the recycled material back into the supply tank 6. If the whole system is operated under superatmospheric pressure, then a constant pressure valve 16 should be provided either in the line 10 or at the outlet of the filter 12.

The mixer 5 may be any one of a number of high speed mixers in which the materials to be mixed are brought into contact in a highly turbulent zone, whereby efficient dispersion is obtained by means of the shearing action of rapidly moving parts alone or combined with the directioning effect of one or more small vanes or baffles. The preferred type of mixer is a centrifugal pump or a mixer known in the industry as the Lancaster disperser. This latter machine consists essentially of a horizontal operating tube with duplex dispersing mechanism each consisting of two impellers attached to the shaft with a set of stator vanes held by tubed wall between impellers. The impellers of one mechanism are of opposite hand to those of the other set, so that the thrust of the two sets of impellers is therefore counterbalanced, practically eliminating end thrust load on the bearings. The radial length of the impeller blades is relatively small so that all the material processed is confined to a thin annular ring where it is subjected to a high average peripheral velocity.

When operating, the machine is entirely filled with material. The dispersing mechanisms force the material outwardly to the ends of the operating tube and up the end passages to the disperser top. The streams enter the central portion of the top tangentially causing them to be intimately mixed as they return down the vertical passage to the middle of the operating tube and again enter the dispersing zones to complete recirculation. New feed is added at both sides of the operating tube on to the faces of the first impellers of each set. Finished product, equal in amount to the new feed, is automatically discharged from the center of the disperser top, this material being an intimate mixture of the product from each dispersing mechanism.

The effective dispersion obtained in the machine is largely due to the manner in which each ingredient is fed and initially dispersed into a separate stream of mother liquor formed by the space between adjacent blades of the impeller. The streams of fortified product on one side and diluted product on the other are intimately mixed and thoroughly dispersed as they progress through the highly turbulent zones of the dispersing mechanisms. An important feature of this duplex design is that the shaft passes through the body of the machine on the discharge side of the operating mechanism only, thus eliminating any possibility of air infiltration. The disperser is a high speed machine, the shaft running usually at a speed of 3600 revs. per min.

A number of dispersers or gear pump equipment similar to colloid mills are available on the market, for example Lancaster dispersers of Model A, series 40 and series 50 or Cornell homogenizers. However, any mixer or high speed homogenizer may be used. These are the ones which bring the various constituents of the lubricants to be prepared into intimate contact for a short interval. With such high speed mixing, a time of 30 seconds is generally more than sufficient for efficient mixing and substantially completes saponification.

According to the preferred method of carrying out the invention, the solution of saponifiable material in the liquid medium, stored in supply tank 1, is preferably manufactured continuously by continuously dissolving a suitable fat or fatty acid in controlled proportions in the desired liquid medium with the application of heat as required; this heating and dissolving may be effected continuously by metering the molten fat or fatty acid from a heated storage tank into the liquid medium. Likewise the dispersion of saponifying agent in liquid medium as stored in supply tank 6 may be manufactured continuously by continuously feeding the desired saponifying agent, as by means of a screw conveyor in controlled proportions into the liquid medium with continuous agitation and with heat if and to the extent necessary.

If optional addition agents are used which are oil-soluble, such as polyisobutylene thickener, as well as various dyes or anti-oxidants, etc., they

are preferably dissolved in the oil which is used for dissolving the saponifiable material, although if preferred, such materials could be dissolved in the liquid medium used for dispersing the saponifying agent.

If as previously suggested, it is desirable first to make a soap-thickened concentrate and then later add more liquid medium (either of the same kind or of a different kind), the additional amount of liquid medium may be fed into the system illustrated in the drawing, from a suitable source, not shown, through the line 17 and a suitable mixer 18 should then be connected into the system in order to insure thorough mixing after the addition of the extra liquid medium through line 17.

In order to further illustrate the operation and advantages of the invention, the following examples are given:

Example 1

Formula:

	Per cent
Hydrogenated fish oil acids	18.0
Hydrated lime	2.64
Low pour Coastal lubricating oil	70.50
Paratac (polybutene solution)	7.85
Zinc naphthenates	0.50
Phenyl alpha naphthylamine	0.50

The molten fish oil acids and 75% of the mineral oil are blended together at 120° F. To this blend is added the zinc naphthenate and polybutene solution (about 8% of polybutene having about 80,000 average molecular weight dissolved in light lubricating oil). The lime is blended in the form of a slurry into the balance of the mineral oil in which the phenyl alpha naphthylamine has been previously dissolved. The separate blends are metered into the disperser or mixing pump and then passed into a screw conveyor when the material is chilled to room temperature and packaged.

Example 2

Formula:

	Per cent
Hydrogenated fish oil acids (Hydrofol)	3.50
Rosin oil	6.00
Sodium hydroxide in a 10% dispersion in gasoline	8.75
Rosin	5.00
Gasoline	76.75

The rosin oil, rosin, and the Hydrofol acid were dissolved in ¾ of the gasoline. In another tank the sodium hydroxide in a very fine powder was dispersed in the gasoline. These two mixtures were then metered into the mixing pump or disperser and then packaged, the finished material reaching its final set in the shipping container.

It is not intended that this invention be limited to any of the particular examples which are given merely for the sake of illustration but only by the appended claims in which it is intended to claim all novelty inherent in the invention as well as various modifications coming within the scope and spirit of the invention.

We claim:

1. Continuous process for making soap-thickened compositions which comprises continuously mixing together at a temperature at least as high as saponification temperature proportioned amounts of a non-aqueous liquid medium containing a saponifiable material dissolved therein and a non-aqueous liquid medium non-reactive with a saponifying agent containing a dry saponifying agent dispersed therein, said mixing being effected in a high speed mixer in which said two liquids are contacted with each other in a highly turbulent zone whereby efficient dispersion thereof is obtained by means of the shearing action of rapidly moving parts.

2. Process according to claim 1 in which the mixture of liquids in the high speed mixer has a temperature at least as high as the saponification temperature and in which the time of actual mixing is not substantially more than about 30 seconds.

3. Process according to claim 1 in which each of the two liquids is preheated to saponification temperature.

4. Process according to claim 1 in which a hydrocarbon liquid is used as the liquid medium both for dissolving the saponifiable material and for dispersing the saponifying agent.

5. Process according to claim 1 in which the high speed mixer used is one of the Lancaster disperser type.

6. Continuous process for making lubricating grease which comprises continuously mixing together at about saponification temperature proportioned amounts of a mineral lubricating oil containing dissolved therein a saponifiable fatty material, and a mineral lubricating oil containing a substantially dry saponifying agent dispersed therein, said mixing being effected in a high speed mixer in which said two liquids are contacted with each other in a highly turbulent zone whereby efficient dispersion thereof is obtained by means of the shearing action of rapidly moving parts.

7. Process according to claim 6 in which the lubricating oil fed into the high speed mixer with the saponifiable material and the saponifying agent constitutes substantially all of the oil present in the finished lubricating grease.

8. Process according to claim 6 in which only a part of the lubricating oil to be present in the finished grease is used in preparing the solution of saponifiable material and the dispersion of saponifying agent which are mixed in the high speed mixer, and the remainder of lubricating oil is blended into the mixture after saponification has been completed.

9. Continuous process for making lubricating grease which comprises continuously mixing together at about 120° F. to 160° F. in a Lancaster disperser proportioned amounts of an aromatic-free coastal lubricating oil containing hydrogenated fish oil acids dissolved therein, and another portion of aromatic-free coastal lubricating oil containing substantially dry hydrated lime dispersed therein.

10. Continuous process for making a soap-thickened composition which comprises continuously dissolving a saponifiable material in controlled proportions in a non-aqueous liquid medium, continuously dispersing a saponifying agent in controlled proportions in another portion of a non-aqueous liquid medium non reactive with said saponifying agent, and continuously mixing the resultant solution and dispersion in controlled proportions at a temperature at least as high as saponification temperature, said mixing being effected in a high speed mixer in which said two liquids are contacted with each other in a highly turbulent zone whereby efficient dispersion thereof is obtained by means of the shearing action of rapidly moving parts.

11. Continuous process for making a soap-

thickened solid fuel composition which comprises continuously mixing together at a temperature at least as high as saponification temperature one portion of an inflammable hydrocarbon liquid containing a soap forming fatty acid dissolved therein, and another portion of an inflammable hydrocarbon liquid containing a dry alkali metal hydroxide dispersed therein, said mixing being effected in a high speed mixer in which said two liquids are contacted with each other in a highly turbulent zone whereby efficient dispersion thereof is obtained by means of the shearing action of rapidly moving parts.

JOHN C. ZIMMER.
ARNOLD J. MORWAY.