

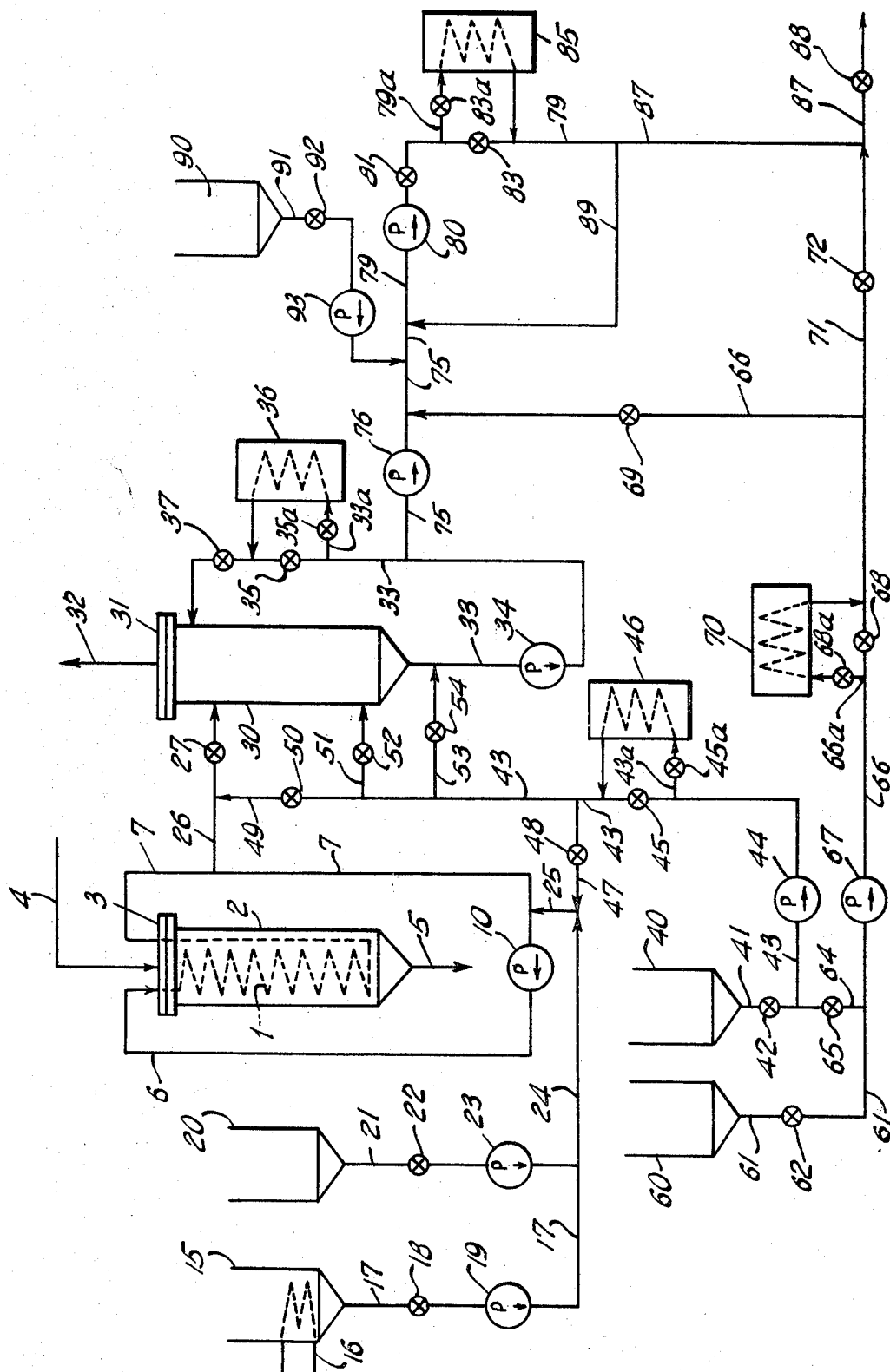
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METHOD AND APPARATUS FOR CONTINUOUS GREASE MANUFACTURE

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METHOD AND APPARATUS FOR CONTINUOUS GREASE MANUFACTURE

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ABSTRACT OF THE DISCLOSURE

Continuous process and apparatus for the preparation of soap thickened grease compositions including the steps of saponification and dehydration, wherein a saponifiable material and a metal base are continuously introduced at an elevated temperature into a saponification zone maintained at superatmospheric pressure and saponified under turbulent conditions characterized by a Reynolds number of at least 4000, the saponified product is blended with lubricating oil to provide a grease mixture containing at least 10% oil, the grease is dehydrated at an elevated temperature and subatmospheric pressure, and any additional oil required is added thereto to form a grease of the desired grade.

This invention relates to an improved method and apparatus for the manufacture of soap thickened greases. More particularly, it relates to an improved method and apparatus for carrying out the preparation of soap thickened greases in a continuous operation including a continuous saponification step.

Continuous processes of various types which have been proposed heretofore for the manufacture of lubricating greases have not been employed successfully in actual grease plant operations, although semicontinuous processes, employing preformed soaps, have been employed fairly successfully in the manufacture of certain greases. The failure of the prior art to provide a successful wholly continuous grease making process is due primarily to the criticality of the process conditions during the steps of the process wherein the soap fibers are principally formed, and the difficulty in carrying out these steps rapidly in a continuous process so as to obtain both an economically feasible throughput rate and satisfactory yields and product quality. In addition, the apparatus designed for carrying out the continuous processes of the prior art are generally lacking in flexibility, and are therefore not capable of producing greases of different types requiring very different process conditions. Equipment of the type required for a continuous grease making operation which can be employed only in the production of a grease of one particular type is ordinarily not economically justified in grease plant operations. For these reasons, continuous grease making processes are not employed commercially in spite of the very considerable inherent advantages of continuous over batch operations generally.

The present invention provides a convenient and flexible method and apparatus for the manufacture of lubricating greases in a continuous operation under conditions which are readily controllable so as to produce optimum yields and product quality. As a particularly advantageous embodiment, it provides a small, compact and inexpensive apparatus capable of producing soap thickened greases of various different types of superior quality in high yields and with extremely high throughput rates, resulting in a production capacity equivalent to that of a large scale commercial grease kettle or other conven-

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tional grease making equipment. An apparatus of this type which has been operated very successfully in the manufacture of various greases occupies a floor space of only 8 x 11 feet x 8 feet in height, and has a throughput rate up to about 2,000 pounds per hour. This capacity is substantially equivalent to that of a large grease kettle, requiring a two-story building for housing, taking into consideration the filling and waiting times required in the operation of the latter. Such an apparatus can be economically constructed and operated at locations where grease is required in large quantities, or it can be truck-mounted for transportation between such locations, thus obviating expensive packaging or bulk shipments, which require specially equipped tankcars.

The grease making process of this invention comprises saponification, dehydration and soap conditioning steps carried out in a continuous manner with recycling of the grease mixture through a shear valve during the soap conditioning step. In the preferred embodiments of the invention, the saponification step is carried out in such a manner that a high degree of turbulence is maintained in the saponification mixture during the reaction. The process also preferably comprises a cooling and finishing step, which may be carried out by the addition of lubricating oil at a lower temperature than the grease mixture or by passing the grease mixture through a cooler. Very advantageously, it may be carried out by a combination of these two methods, and with shearing of the grease mixture within a suitable temperature range either during or after the cooling.

The apparatus which the invention provides for carrying out this grease making process comprises a single zone for the dehydration and soap conditioning steps, provided with a recycle line containing a shear valve for recycling the grease mixture from the bottom to the top of the zone with shearing of the recycle stream, and adapted to be operated at a substantially lower pressure than the saponification zone. The saponification zone is preferably a flow type reactor, very suitably comprising a section of pipe having an inside diameter of about 0.5–1.25 inches, and preferably provided with a recycle system for recycling the saponification mixture through the reactor at a sufficient rate to maintain turbulent flow. The apparatus preferably comprises a finishing section also, with provisions for cooling the grease mixture either by the addition of cold lubricating oil or by passing it through a heat exchanger. An apparatus representing a particularly preferred embodiment of the invention comprises a finishing section wherein the grease mixture may be recycled through a shearing means, such as a shear valve, either during or after this cooling.

In carrying out the process under the preferred conditions, the saponification zone is maintained at an elevated temperature and pressure at least sufficient to maintain the water present in the saponification mixture in the liquid phase, and the dehydration zone is operated at an elevated temperature below the melting point of the soap and under a substantially lower pressure than the saponification zone, so that the major portion of the water is flashed off when the grease mixture enters the dehydration zone. Any remaining water is removed during recycling of the grease mixture through the shear valve, which in effect subjects the grease mixture to a continuous flashing operation by pressure release of the recycle stream through the valve. The recycling is preferably carried out at a rapid rate, such that the grease mixture is subjected to multiple passes through the shear valve operated with at least a substantial pressure drop during the residence time of the grease mixture within the zone. Cooling of the grease mixture is preferably carried out with the addition of lubricating oil at a substan-

tially lower temperature than the grease mixture, and very advantageously in some cases with recycling of the grease mixture through a cooler. The process is also carried out very advantageously in some cases with recycling of the grease mixture through a shearing means during or after the cooling at a temperature within a range which will vary somewhat with the different greases, depending chiefly upon the character of the soap thickener.

Greases of excellent quality are obtained in the above manner in good yields and in greatly reduced manufacturing times as compared with the prior art processes, due in a large measure to the effect upon the soap fiber development of the recycle shearing during the dehydration and soap conditioning steps, and also to the combined effect of this shearing with the effect of carrying out the saponification under turbulent conditions, which provides a grease mixture wherein the soap molecules or micelles are present in a highly dispersed state.

The invention will be better understood from the following description in conjunction with the accompanying drawing, which is a schematic diagram of an apparatus representing a preferred embodiment of the invention.

Referring to the drawing, 1 is a tubular reactor in the form of a coil, situated inside chamber 2 and connected with lines 6 and 7. Chamber 2 is sealed by flange 3 and provided with inlet line 4 and outlet line 5 for steam or other heating fluid. Vessel 15 contains saponifiable material, or a mixture of saponifiable material and lubricating oil, which is maintained at a temperature above the melting point of the saponifiable material by heating means 16. Ordinarily, vessel 15 will contain a mixture of saponifiable material and lubricating oil comprising at least 10 percent by weight of the mixture. It is generally preferred to employ a mixture comprising about 20 to 60 percent by weight of saponifiable material, although lower amounts down to about 5 percent by weight and also higher amounts up to 100 percent by weight of saponifiable material may be employed in some cases. Vessel 20 contains a water solution or oil slurry of metal base.

In carrying out the grease making process, the saponifiable material passes at a controlled rate through line 17 containing valve 18 and pump 19 into line 24, while a solution or slurry of metal base passes from tank 20 at a controlled rate through line 21 containing valve 22 and pump 23 into line 24. From line 24 the mixture of saponifiable material and metal base passes through line 25 into line 7 at the intake of pump 10, and from pump 10 it passes through line 6 into zone 1. When the saponification is carried out employing a slurry of the metal base in oil, it is generally desirable to introduce a small amount of water or steam into the reaction zone in order to promote the reaction. The reaction mixture in reaction zone 1 is maintained under superatmospheric pressure at least sufficient to maintain the water present or produced in the reaction in the liquid phase, and at an elevated temperature sufficient to obtain a rapid reaction between the metal base and the saponifiable material. Suitable reaction conditions include broadly pressures in the range from about 10 to about 300 pounds per square inch gauge and temperatures from about 180° F. up to above the melting point of the soap formed in the reaction. The preferred conditions include pressures in the range from about 50 pounds per square inch to about 200 pounds per square inch and temperatures in the range from about 200° F. to about 350° F.

The reactant stream is passed through reaction zone 1 at a velocity which is preferably sufficient to maintain turbulent flow within the zone. With special advantage, the reactant stream may be passed through the reaction zone at a velocity resulting in highly turbulent flow, preferably at a velocity resulting in a Reynolds number in the range from about 4,000 to about 100,000. Flow rates required to obtain the desired degree of turbulence are generally within the range from about 0.6 to about 12.0 cubic feet per minute per square inch of reactor cross

section. In the process comprising the preferred embodiment of this invention, the saponification mixture is recycled continuously through reactor 1 by way of lines 6 and 7 and pump 10, as a means of obtaining a sufficiently high rate of flow of the reactant stream through the saponification zone. In this manner, a high rate of flow through reaction zone 1 is maintained which is not dependent upon the feed rate, and turbulent flow through the reaction zone can therefore be maintained even with a saponification mixture requiring a relatively long residence time for substantially complete reaction or where an input rate below that required for turbulent flow is required by other conditions of the process. The recycle rate employed is ordinarily in the ratio from about 10:1 to about 100:1 with the rate of throughput, although somewhat lower or higher recycling ratio may be employed in some cases, such as recycle ratio as low as about 1:1 and as high as about 200:1.

Saponification products obtained under the above conditions are especially suitable for use in the subsequent grease making steps of our process because of the readiness with which they accept additional lubricating oil and the shorter soap conditioning periods which they require as compared with grease mixtures obtained under other saponification conditions. The different physical conditions of these products are shown by the fact that they form grease-like products immediately upon cooling when the saponification mixture contains lubricating oil, differently from saponification products obtained under different conditions including such products obtained in reactors where turbulence is maintained by other means. This different result is presumably due to the severe shearing to which the reaction mixture is subjected when it is passed through a relatively small diameter tube under turbulent flow conditions.

A product stream from reaction zone 1 passes to dehydration zone 30 by way of line 7 and line 26 containing valve 27. Dehydration zone 30 is an upright zone of relatively large diameter as compared with zone 1, sealed by flange 31 and provided with line 32 which is connected to a pressure regulator means (not shown). It may be jacketed or otherwise provided with indirect heating or cooling means. The grease mixture in zone 30 is maintained at an elevated temperature above about 225° F. and ordinarily at least about 250° F., but below the melting point of the soap present in the grease mixture, and at a pressure substantially lower than that in reaction zone 1, very suitably under a partial vacuum of from about 5 to about 25 inches of mercury. In the preparation of lithium 12-hydroxystearate thickened greases, the grease mixture in zone 30 is preferably maintained at a temperature in the range from about 350° F. to about 375° F.

During its residence in zone 30, the grease mixture is recycled continuously through line 33, containing pump 34 and valves 35 and 37. Valve 37 is a shear valve, suitably a gate valve, set in a partially closed position so as to give a pressure drop of about 10–200 pounds per square inch, and preferably about 25–125 pounds per square inch across the valve. The recycling is preferably carried out at a rapid rate, such that the volume of recycled grease mixture is equal to the total average volume of grease mixture within zone 30 (1 batch turnover) within one minute, and sufficient to provide at least about 5 batch turnovers, and most advantageously at least 10 batch turnovers, during the average residence time of the grease mixture within the zone. The grease residence time in zone 30 may be only sufficient to obtain substantially complete dehydration of the grease mixture, the soap conditioning step in this case taking place simultaneously with the final dehydration stages. It is ordinarily prolonged somewhat so as to provide an additional soap conditioning period, preferably for at least about 5 minutes, particularly when the dehydration is accomplished substantially entirely in the initial flashing opera-

tion. In carrying out the process under the preferred conditions, the residence time of the grease mixture in zone 30 may be from a few minutes up to about 1 hour, depending chiefly upon the character of the soap in the grease mixture, and to a less extent upon other factors such as temperature, soap concentration of the grease mixture and character of the lubricating oil. In the preparation of lithium and calcium hydroxy fatty acid soap thickened greases, a suitable residence time of the grease mixture in zone 30 will usually be from about 5 to about 20 minutes, although somewhat shorter or longer periods may be employed in some cases.

Additional lubricating oil from tank 40 may be added to the grease mixture at various steps in the process in order to obtain the desired soap concentration or to assist in heating or cooling the grease mixture. This additional oil may pass into the grease mixture in line 26 by way of line 41, containing valve 42, line 43 containing pump 44 and valve 45, and line 49 containing valve 50. Additional lubricating oil is added very advantageously in this manner so as to provide at least about 10 percent of lubricating oil in the grease mixture in line 26 when the saponification is carried out with no lubricating oil or only a small amount of lubricating oil present in the saponification mixture. Such oil addition may also be employed as a means of heating the grease mixture in order to increase the water removal when the grease mixture is flashed into zone 30. The oil added for this purpose is preheated by passing through heater 46 by way of line 43a, containing valve 45a.

Additionally or alternatively, lubricating oil from tank 40 may pass into the saponification mixture in zone 1 by passing from line 43 through line 47 containing valve 48 and into line 25. Oil added in this manner is employed either in addition to or in place of lubricating oil employed in admixture with the saponifiable material in tank 15. It is preferably preheated in heater 46, very suitably to a temperature in about the range 150–350° F.

Additionally or alternatively to the lubricating oil addition in the above manner, lubricating oil may be added from tank 40 to the grease mixture during the soap conditioning step. The oil added at this stage of the process may pass into the grease mixture in zone 30 through line 51 containing valve 52. With special advantage in some cases, it may pass through line 53 containing valve 54, into the recycle stream of grease mixture in line 33 as the means of aiding in the recycling when a heavy grease mixture is being circulated, and also as a means of increasing the rate of dehydration by increasing the temperature of the recycle stream in some cases. Additional oil is added in the above manner as required to provide a grease mixture in zone 30 containing at least about 25 percent by weight of lubricating oil, and preferably at least about 40 percent by weight of lubricating oil.

In addition to the function of the lubricating oil addition to the grease mixture in zone 30 as a means of obtaining the desired soap concentration, the oil addition may be employed as a means of either heating or cooling the grease mixture to a temperature within the desired soap conditioning temperature range. When the saponification is carried out at a temperature above the melting point of the soap, the oil added to the grease mixture in zone 30 by way of lines 51 or 53 is preferably at a lower temperature than the grease mixture leaving the saponification zone. When the saponification is carried out at a temperature below the desired temperature range for the soap conditioning treatment, the lubricating oil added as described above is preferably preheated. The temperature of the lubricating oil and the amount added may be adjusted so as to give the desired soap concentration in the grease mixture in zone 30 and also to provide a temperature within the desired soap conditioning temperature range.

Indirect heating or cooling of the grease mixture in

zone 30 may be employed either in addition or alternatively to the heating or cooling obtained by oil addition as described above. The indirect heating or cooling may be obtained very conveniently by employing a jacketed vessel for zone 30 and passing a heat exchange fluid through the vessel jacket. Very advantageously in some cases, the indirect heating or cooling is applied to the recycle stream of grease mixture in line 33 by passing the grease mixture through heat exchanger 36 by way of line 33a, containing valve 35a. It is, for example, particularly advantageous in some cases to cool the recycle stream in this manner in order to obtain an increased effect by the shearing, due to the more viscous grease mixture obtained by the cooling.

A stream of substantially dehydrated grease mixture is continuously withdrawn from the recycle stream in line 33 by way of line 75 containing pump 76. Additional lubricating oil may be added to the grease mixture in line 75 from tank 60 by way of line 61 containing valve 62 and line 66 containing pump 67 and valve 68. It is ordinarily preferable to add this oil at a temperature substantially lower than that of the grease mixture, very suitably in some cases at ambient temperature. However, in many cases it is advantageous to preheat the oil by passing it through heat exchanger 70 by way of line 66a, containing valve 68a, particularly where a high rate of oil addition is employed or where it is desirable to employ a slower cooling rate. Alternatively or additionally, oil may be introduced into line 75 from tank 40 by way of line 41, line 64, containing valve 65, and line 66. Tank 60 is generally employed in conjunction with tank 40 in the process only when two different lubricating oils are employed in the grease.

The additional oil added to the grease mixture in line 75 may amount to as much as about 90 percent by weight of the total oil in the finished grease. It is ordinarily preferable to carry out the grease preparation with about 20–80 percent by weight of the total oil contained in the grease added in this manner at a temperature at least about 100° F. below the temperature of the grease mixture in line 75.

From line 75 the grease mixture passes into line 79, containing pump 80 and valves 81 and 83. Valve 81 is a shear valve, such as a gate valve, which may be operated with a substantial pressure drop. Where it is desirable to obtain additional cooling, the grease mixture may pass through cooler 85, by way of line 79a, containing valve 83a. Recycling of the grease mixture through line 79 by way of line 89 may be employed in order to obtain multiple passes through shearing valve 81 and cooler 85. From line 79 a stream of the grease mixture is taken off through line 87, containing valve 88. Additional lubricating oil at the same or a lower temperature may be added to this stream of grease mixture by way of line 71, containing valve 72, either alternatively or in addition to the lubricating oil added through line 66 as described above. When additional oil is added in this manner it becomes mixed with the grease mixture as the combined stream passes through valve 88, which is operated as a shear valve. The stream of grease mixture containing oil added in this manner may pass through additional shearing means if desired.

Any additives employed in the grease are preferably introduced into the grease mixture during the cooling, ordinarily where the grease mixture is below about 250° F. As shown in the diagram, the additives may be added from tank 90 by way of line 91 containing valve 92 and pump 93 into the grease mixture in line 75 after the addition of lubricating oil from line 66. When the grease mixture at this point is not at a suitably low temperature, the additives may be added at some later point in the cooling and finishing section. In the manufacture of cup greases, the water of stabilization is also added during the finishing when the grease mixture is at a temperature below 210° F.

It is ordinarily advantageous to recycle the grease mixture through shear valve 81 operated with a pressure drop in about the range 20–200 pounds per square inch gauge, employing a recycle ratio from about 1:1 to about 100:1 and preferably from about 5:1 to about 50:1. Shearing in this manner is preferably carried out upon the grease mixture at a temperature below about 300° F., and most advantageously in most cases at a temperature within the range from about 250° F. to about 150° F.

The metal base employed in the saponification may be a hydroxide or other suitable basic reacting compound of any of the metals ordinarily employed as the metal component of the soap in the preparation of lubricating greases, such as sodium, lithium, potassium, calcium, barium, magnesium, zinc, cobalt, manganese aluminum, lead, etc., as well as mixtures of two or more metals. It is preferably a metal oxide, hydroxide or carbonate. The greases which are most advantageously prepared by the method of this invention are those wherein the soap thickener is an alkali metal or alkaline earth metal soap, or a mixture of two or more soaps of these classes.

Suitable saponifiable materials for use in these grease preparations comprise higher fatty acids containing from about 12 to 32 carbon atoms per molecule and hydroxy substituted higher fatty acids, their glycerides and other esters and mixtures thereof. The invention also contemplates grease preparations carried out in the manner described above wherein such higher fatty acid materials are employed in conjunction with lower fatty acid materials, such as fatty acids containing from one to about 6 carbon atoms per molecule, their glycerides and other esters. Such lower fatty acid materials may be employed in amounts giving a mol ratio of lower fatty acid to higher fatty acid from below 1:1 up to about 20:1, respectively. Also, intermediate fatty acid materials may be employed in conjunction with the higher fatty acid materials in varying amounts, ordinarily in amounts giving a mol ratio with the higher fatty acid material below about 1:1, respectively.

The oleaginous liquids employed in these greases may be any suitable oils having lubricating characteristics, including the conventional mineral lubricating oils, synthetic oils obtained by various refining processes such as cracking and polymerization and other synthetic oleaginous compounds such as high molecular weight ethers and esters. The dicarboxylic acid esters, such as di-2-ethylhexyl sebacate, di(secondary amyl)sebacate, di-2-ethylhexyl azelate, diisooctyl adipate, etc., comprise a particularly suitable class of synthetic oils and may be employed either as the sole oleaginous component of the grease or in combination with other synthetic oils or mineral oils. Suitable mineral oils for use in these greases are those having viscosities in the range from about 100 to about 8000 seconds Saybolt Universal at 100° F., which may be blends of low and high viscosity oils. They may be either naphthenic or paraffinic in type, or blends of two or more oils of these different types.

In the production of greases from synthetic oils which are hydrolyzed under the saponification conditions, the saponification is preferably carried out in the absence of any lubricating oil or of a minor amount of a lubricating oil which is substantially inert under the saponification conditions such as a mineral oil and the synthetic oil added at later stages of the grease making process as described hereinabove.

The following examples are illustrative of lubricating grease preparations carried out in accordance with this invention.

EXAMPLE I

A lithium 12-hydroxystearate thickened grease was prepared by the method of this invention as described below.

The apparatus employed in the preparation comprised a coil reactor, a dehydrator and a cooler, with auxiliary

equipment for circulating the grease mixture through each of these zones as shown in the figure, as well as for introducing reactants and additional lubricating oil, moving the grease mixture between the zones and withdrawing a product stream. The reactor consisted of a 21-foot section of ¾ inch Schedule 40 black iron pipe (0.824 inch inside diameter) formed into a coil having a 4 inch inside diameter and mounted in a 3 foot section of 12 inch pipe serving as a steam chamber. The recycle line on the reaction zone comprised 9 feet of ¾ inch pipe containing a recycle pump, which was a Viking Rotary heavy duty pump having a capacity of 18 gallons per minute at 1200 revolutions per minute. The capacity of the reactor and recycle line including the pump was 0.12 cubic foot. The dehydrator comprised a 3 foot section of 10 inch Schedule 40 black iron pipe with a blind flange and a bell cap on the ends and electrically heated by a wrapping of resistance wire on the outside. The recycle line on the dehydrator consisted of a 5 foot section of 0.824 inch inside diameter pipe containing a recycle pump of the same type as that employed in the recycle line on the reactor and a Fulflo valve. The dehydrator was connected at the top by a 2.067 inch inside diameter pipe to a vacuum jet with a condenser. The reactor and dehydrator were connected by a 0.824 inch inside diameter pipe containing Fulflo valve. The cooler was a watercooled Graham Heliflow shell and tube type exchanger, through which the grease mixture was circulated at 18 gallons per minute, resulting in turbulent flow.

The following materials were employed in the grease preparation. The saponifiable material employed was a commercial 12-hydroxystearic acid, having a saponification number of 186, a neutralization number of 177 and an iodine number of 3. The lubricating oil was a blend having a Saybolt Universal viscosity at 210° F. of about 84 seconds, obtained by blending 35 percent by volume of a paraffinic distillate oil, having a Saybolt Universal viscosity at 100° F. in the range 175–190 seconds, with a steam refined and deasphalted Manvel residuum having a Saybolt Universal viscosity at 210° F. in the range 155–163 seconds. The saponifying agent employed was an aqueous lithium hydroxide solution comprising 9.3 percent of lithium hydroxide.

Following is a detailed description of the procedure employed in this preparation. A solution comprising 14 percent by weight of the 12-hydroxystearic acid in lubricating oil preheated in the charge tank to 203° F. was charged to the reactor at a rate of 86.2 pounds per hour and the aqueous lithium hydroxide solution at room temperature was charged to the reactor at a rate of 12.8 pounds per hour. The mixture in the reaction zone was heated to 310° F. under a pressure of 70 pounds per square inch at the reactor coil inlet and of 68 pounds per square inch at the reactor coil outlet. The recycle pump was operated at 18 gallons per minute, resulting in a recycle ratio of 80:1. A product stream was taken from the recycle stream and passed with pressure release through the back pressure valve to the dehydrator, where it was heated to 365° F. under a vacuum of 15 inches of mercury. The grease mixture in the dehydration zone was recycled at a rate of 18 gallons per minute with the valve in the recycle line set to give a pressure drop of about 67 pounds per square inch. Additional lubricating oil preheated in the storage tank to 344° F. was introduced into the recycle stream at the inlet side of the recycle pump at a rate of 161.8 pounds per hour.

The volume of grease mixture in the dehydration zone was maintained at about 0.84 cubic foot, which resulted in an average residence time of 10 minutes for the grease mixture in the dehydration zone, during which time it was recycled through the recycle line an average of 28.6 times, i.e., an average of 28.6 turnovers of grease mixture during the average residence time.

A product stream was taken from the recycle stream

at the discharge side of the recycle pump and recycled through the cooler until it was cooled to 200° F.

A smooth grease of excellent texture and appearance was obtained as described above in a total preparation time of 15.3 minutes. The following analyses and test results were obtained upon this grease.

Composition, percent:

Li 12-hydroxystearate	6.0
Free neutral fat	0.41
Free fatty acid	None
Free alkali	0.12
Water	0.05

Tests:

Penetration, ASTM at 77° F.—	
Unworked	272
Worked, 60 strokes	281
Worked, 10,000 strokes	287
Dropping point, ASTM, ° F.	380
Bomb oxidation, ASTM (100 hrs at 210° F. pressure drop, p.s.i.)	8

Shell roll test—

Original penetration	265
Rolled penetration	283

Points increase 18

The above data show that a very satisfactory grease was made with a total average residence time in the continuous grease making unit of only 15.3 minutes. This compares with an average time of about 11.4 hours required for commercial preparation of a lithium 12-hydroxystearate grease by a conventional low temperature process. The grease was furthermore obtained in optimum yield, as shown by the fact that a portion of the grease which was subjected to milling with one pass through a Premier colloid mill at 0.003 inch clearance had a worked penetration of 284, or substantially the same as the unmilled product.

EXAMPLE II

A calcium 12-hydroxystearate thickened grease was prepared by the method of this invention as described below.

The apparatus employed was the same as that described in Example I.

The following materials were employed in the preparation. The saponifiable material was a commercial 12-hydroxystearic acid as described in Example I. The mineral oil employed was a paraffinic distillate oil having a Saybolt Universal viscosity at 100° F. of about 347 seconds and a small amount, equal to about 1 percent of the finished grease of a Manvel residuum having a Saybolt Universal viscosity at 210° F. of 55 seconds. The saponifying agent was lime employed in the form of a 10 percent slurry in lubricating oil.

Following is a detailed description of the method employed in the preparation. A 20.3 percent solution of the saponifiable material in lubricating oil preheated to 200° F. was charged to the reaction zone at a rate of 87.6 pounds per hour and the slurry of lime in lubricating oil at room temperature was charged at a rate of 23.8 pounds per hour. The reaction mixture in the reaction zone was heated to 261° F. under a pressure of 70 pounds per square inch (coil inlet) to 50 pounds per square inch (coil outlet). Recycling on the reactor was carried out at 18 gallons per minute, resulting in a recycle ratio of 66:1. The product stream, having a calculated soap content of 17.2 percent, was introduced into the dehydrator where it was maintained at 282° F. and under a vacuum of 5.0 inches of mercury. The grease mixture was recycled from the bottom to the top of the dehydration zone at a rate of 18 gallons per minute by means of a recycle line containing a recycle pump and a valve operated with a pressure drop of about 63 pounds per square inch. Addi-

tional lubricating oil preheated to 283° F. was introduced into the recycle stream at the inlet side of the recycle pump at a rate of 138 pounds per hour. A volume of about 0.88 cubic foot was maintained in the dehydration zone, which resulted in an average residence time of the grease mixture of 10.7 minutes, during which time it was recirculated through the recycle line for an average of 29.2 times. The product stream from the dehydration

The materials employed were the following. The sapon-gallons per minute, resulting in 30 passes of the reaction mixture through the cooling zone. The grease mixture was cooled in this manner to 195° F.

A smooth buttery grease was obtained in the above manner. The following analyses and test results were obtained upon this grease.

Composition, percent:

Ca 12-hydroxystearate	8.1
Free neutral fat	0.29
Free fatty acid	0.05
Free alkali	None
Water	Trace

Tests:

Penetration, ASTM, at 77° F.—

Unworked	249
Worked	276
Dropping point, ASTM, ° F.	305
Bomb oxidation, ASTM (100 hrs. at 210° F. pressure drop, p.s.i.)	4

The above data show that a grease of satisfactory lubricating properties was obtained in good yield in the preparation, wherein the average overall time was only about 15.3 minutes. In addition, difficulties encountered in prior art processes in obtaining greases of this type in satisfactorily smooth form are overcome by carrying out the preparation in the manner described.

EXAMPLE III

A sodium tallowate grease was prepared by the method of this invention as described below.

The apparatus employed was the same as that employed in the preparation described in Example I except that a shear valve was provided in the finishing section as shown in FIG. 1.

The materials employed were the following. The saponifiable material employed was a commercial high acid hard tallow. Typical tests upon this material include a saponification number of 200, a free fatty acid content of 4.2 percent, an iodine number of 52 and a titer of 41.6° C. The lubricating oil employed was a deasphalted and de-waxed residuum from a paraffin base crude having a Saybolt Universal viscosity at 210° F. of about 189 seconds. The saponifying agent was sodium hydroxide in the form of a 49 percent aqueous solution.

Following is a detailed description of the method employed in the preparation. Streams of saponifiable material and lubricating oil preheated to 210° F. were charged to the saponification zone at rates of 0.296 pound per minute and 0.557 pound per minute, respectively, while the alkali solution at room temperature was charged at 0.086 pound per minute. The reaction mixture was heated in the reactor to 333° F. under a pressure of 155 pounds per square inch (coil inlet) to 110 pounds per square inch (coil outlet). Recycling on the reactor was carried out at a rate of 18 gallons per minute, resulting in a recycle ratio of 144:1. The product stream, having a calculated soap content of 30.8 percent, was introduced into the dehydrator where it was maintained at 364° F. and under a vacuum of 20 inches of mercury while it was recycled at a rate of 18 gallons per minute through the recycle line with the shear valve set to give a pressure drop of 70 pounds per square inch. No additional oil was added to the grease mixture in the dehydrator in this preparation.

The level of the grease mixture in the dehydrator was

maintained at about 15 inches, resulting in an average residence time of the grease mixture of 49.4 minutes, during which time the grease mixture was subjected to an average of 144 passes through the recycle line.

The dehydrated product stream withdrawn from the dehydrator was cooled by the addition of lubricating oil at a lower temperature and recycled through the shear valve with a recycle rate of 10 gallons per minute, resulting in a recycle ratio of 14.8:1 and a total time of the grease mixture in the finishing section of 0.44 minute. Circulation through the cooler was not employed in this preparation. The additional oil at 190° F. was introduced into the stream of grease mixture at a rate of 4.150 pounds per minute. The shear valve in the recycle line was set to give a pressure drop of 140 pounds per square inch. The product stream was withdrawn from the finishing section at 195° F.

A grease having the smooth slightly stringy texture desired in a sodium base grease of this type and superior lubricating properties was obtained as described above. The following analyses and tests were obtained upon this product.

Composition, percent:

Sodium soap -----	5.3
Free neutral fat -----	0.23
Free fatty acids -----	0.17
Free alkali -----	None
Water -----	0.05

Tests:

Dropping point, ASTM, ° F. -----	306
Penetration, ASTM, at 77° F.—	
Unwork -----	355
Work, 60 strokes -----	383
Worked, 100,000 strokes -----	382
Shell roll tests—Penetration point change ---	+12

The above tests show that a sodium tallowate thickened grease was obtained in a good yield and in a greatly shortened manufacturing time by the method of our invention, the total time required by our method being about 1 hour, as compared with over 21 hours required for making a commercial batch of this grease in a conventional grease kettle. In addition, the grease prepared by our process was superior in certain respects, particularly in shear stability, to the conventionally prepared greases, as shown by the fact that it underwent only 12 points penetration change in the Shell roll tests, as compared with 24–65 points penetration change of the conventionally prepared products.

Obviously, many modifications and variations of the invention as hereinbefore set forth may be made without departing from the spirit and scope thereof, and therefore, only such limitation should be imposed as are indicated in the appended claims.

We claim:

1. A continuous process for manufacturing lubricating grease which comprises continuously introducing a reaction mixture consisting essentially of saponifiable material and metal base into a tubular reaction zone wherein the said mixture is maintained at a temperature of 180° F. to above the melting point of the soap formed in the reaction and at a pressure of between 10 and 300 pounds per square inch gauge under turbulent conditions characterized by a Reynolds number of at least 4,000 for a period sufficient to obtain at least substantially complete reaction, continuously withdrawing a product stream from the reaction zone, introducing into the product stream any lubricating oil required to give a grease mixture containing at least about 10 percent of lubricating oil, continuously introducing said grease mixture comprising lubricating oil and soap into a dehydration zone wherein the grease mixture is maintained at an elevated temperature above about 225° F. but below the melting point of the soap and under a pressure substantially lower than the pressure in the reaction zone, maintaining the said grease

mixture in the dehydration zone for a period sufficient to accomplish at least substantially complete dehydration, adding any additional lubricating oil required to provide a grease mixture comprising at least about 25 percent of lubricating oil, continuously withdrawing a product stream from the said dehydration zone, cooling the said withdrawn stream and mixing it with any additional lubricating oil required to provide a grease of the desired grade, said grease mixture in the said dehydration zone being circulated from the bottom to the top of the said dehydration zone through a recycle line containing a shear valve operated with a pressure drop across the said valve of about 10–200 pounds per square inch at a rate such that the volume of recycled grease mixture during the average residence time of the grease mixture within the dehydration zone equals at least about 5 times the total average volume of grease mixture within the said zone.

2. The process of claim 1 wherein the said reaction mixture is passed through the said tubular reaction zone at a rate within the range from about 0.6 to about 12.0 cubic feet per minute per square inch of reactor cross sectional areas.

3. The process of claim 1 wherein preheated oil is added to the said grease mixture within said dehydration zone.

4. The process of claim 1 wherein the said preheated oil is added to the said grease mixture in the said dehydration zone recycle line.

5. The process of claim 1 wherein the said product stream from the said dehydration zone is cooled by the addition of lubricating oil.

6. The process of claim 5 wherein the said product stream from said dehydration zone and any additional lubricating oil are subjected to a shearing operation.

7. The process of claim 1 wherein the said reaction mixture comprises a minor proportion of water.

8. The process of claim 1 wherein the said metal base is selected from the class consisting of oxides, hydroxides and carbonates of alkali metals and alkaline earth metals.

9. A continuous process for manufacturing lubricating greases which comprises continuously introducing a reaction mixture consisting essentially of saponifiable material, lubricating oil comprising at least about 10 percent by weight of the said mixture, metal base, and a minor amount of water, into a reaction zone wherein the said mixture is maintained at an elevated temperature in about the range 200–350° F., under superatmospheric pressure of about 20–200 pounds per square inch and in a highly turbulent condition characterized by a Reynolds number of at least 4,000 for a period sufficient to obtain at least substantially complete reaction between the said saponifiable material and the said metal base, continuously withdrawing a product stream of the grease mixture from the said reaction zone and introducing the said withdrawn stream into a dehydration zone wherein the grease mixture is maintained at an elevated temperature in the range from about 250° F. to just below the melting point of the soap contained in the said grease mixture and under a partial vacuum in the range from about 5 to 25 inches of mercury, maintaining the said grease mixture in the said dehydration zone for a period sufficient to accomplish at least substantial dehydration, adding any additional lubricating oil required to provide a grease mixture comprising at least about 40 percent of lubricating oil, continuously withdrawing a product stream from the said dehydration zone, cooling the said withdrawn stream and mixing it with any additional lubricating oil required to provide a grease of the desired grade, said grease mixture in the said dehydration zone being circulated from the bottom to the top of the said dehydration zone through a recycle line containing a shear valve operated with a pressure drop of about 20–125 pounds per square inch across the said valve at a rate such that the volume of recycled grease mixture during the average residence

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time of the grease mixture in the dehydration zone equals at least about 10 times the total average volume of grease mixture within the said zone.

10. The process of claim 9 wherein the said metal base is selected from the group consisting of oxides, hydroxides and carbonates of alkali metals and alkaline earth metals.

11. The process of claim 1 wherein said reaction mixture comprises a minor portion of lubricating oil.

12. Apparatus for the continuous manufacture of a lubricating grease which comprises in combination, a tubular reaction zone, means for supplying heat to said reaction zone, a dehydration zone of relatively large cross-sectional area in comparison with the cross-sectional area of said reaction zone, means for supplying heat to said dehydration zone, means for continuously introducing into said reaction zone at an elevated pressure saponifiable material and metal base reactants in relative proportions to effect saponification of said reactants at an elevated pressure and temperature under turbulent flow conditions characterized by a Reynolds number of at least 4000, means for continuously discharging saponified reaction product from said reaction zone, means for continuously introducing lubricating oil into said discharged reaction product as required to provide a grease mixture containing at least about 10% by weight of lubricating oil, means for continuously introducing said grease mixture into said dehydration zone maintained at a substantially lower pressure than said reaction zone, means for continuously withdrawing a portion of the resulting dehydrated grease mixture from a lower part of said dehydration zone and passing said withdrawn portion through a restricted orifice effecting shearing thereof and returning said sheared portion to an upper part of said dehydration zone, and means for continuously discharging the resulting sheared grease mixture from said dehydration zone.

13. Apparatus as claimed in claim 12 wherein said reaction zone is provided with means for continuous withdrawal of a portion of said saponified reaction product from said reaction zone and return of said withdrawn portion to said reaction zone.

14. Apparatus as claimed in claim 13 wherein said means for withdrawing a portion of said saponified reaction product from said reaction zone is provided with means for introducing lubricating oil into said withdrawn portion.

15. Apparatus as claimed in claim 12 wherein said dehydration zone is provided with means for introducing additional lubricating oil directly into said dehydration zone.

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16. Apparatus as claimed in claim 12 wherein said means for continuous withdrawal of a portion of the dehydrated grease mixture from said dehydration zone is provided with means for introducing lubricating oil into the said withdrawn portion.

17. Apparatus as claimed in claim 12 wherein said means for continuously discharging the resulting dehydrated grease mixture from said dehydration zone is provided with means for introducing lubricating oil into the discharged mixture.

18. Apparatus as claimed in claim 12 wherein said means for discharging said dehydrated grease mixture from said dehydration zone is provided with a restricted orifice effecting a pressure drop of the order of 20 to 200 p.s.i. to effect shearing of said mixture.

19. Apparatus as claimed in claim 12 including means for cooling said discharged dehydrated grease mixture.

20. Apparatus as claimed in claim 19 wherein said cooling means comprises means for introducing lubricating oil into said sheared mixture.

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252—35, 41

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,475,335

Dated October 28, 1969

Inventor(s) John H. Greene, William R. Hencke,
Clarence L. Dowden, Jr. and Herbert J. Pitman

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

COLUMN 1, before line 30, insert as a separate paragraph
--This application is a continuation of application Serial
No. 333,164 filed Dec. 24, 1963 and now abandoned.--
COLUMN 9, line 53, "residum" should read --residuum--.
COLUMN 10, line 9, delete "The materials employed were the
following. The sapon-" and substitute --zone was recycled
through the cooler at a rate of 18--. COLUMN 12, line 23,
"areas" should read --area--.

SIGNED AND
SEALED
AUG 25 1970

(SEAL)

Attest:

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Attesting Officer

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Commissioner of Patents