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(54) Process for the continuous manufacture of a lubricating grease.

(57) A process for the continuous manufacture of a lubricating grease comprising blending feed materials and lubricating oil and pumping the resulting blend through at least one motionless mixer which is heated to such a temperature that soap formation takes place and a resultant grease is formed.

PROCESS FOR THE CONTINUOUS MANUFACTURE OF A
LUBRICATING GREASE

This invention relates to a process for the continuous manufacture of a lubricating grease and in particular of a base grease which serves as a basis for the manufacture of a wide variety of lubricating grease formulations containing one or more
5 additives. By first making a base grease a great flexibility is obtained in making a great variety of grease formulations using the same grease manufacturing basic unit.

From European patent application 0072184 a process for the continuous manufacture of a lubricating grease, which process
10 shows a great flexibility, is already known. According to this process a rotating screw process unit is used.

It has now been found that such processes, which enable the manufacture of a great variety of greases, such as the greases described in said European patent application, can be carried out
15 in a simpler way without the use of moving parts, namely by using motionless mixers, which are readily commercially available.

Therefore the present invention relates to a process for the continuous manufacture of a lubricating grease comprising blending feed materials and lubricating oil and pumping the resulting blend
20 through at least one motionless mixer which is heated to such a temperature that soap formation takes place and a resultant grease is formed.

Suitable feed materials are the usual thickening or gelling agents which in the case of soaps are often made in situ in at
25 least part of the lubricating oil from saponifiable material, saponification agents and optionally complexing agents.

Suitable saponifiable materials are long chain fatty esters, especially glycerides, in particular where the fatty group contains

a hydroxy radical, such as ricinoleyl (castor oil) or hydrogenated ricinoleyl (hydrogenated castor oil).

Another method of soap formation is to neutralize fatty acids or hydroxy fatty acids in mineral oil with alkali metal or alkaline earth metal hydroxides (or mixtures of both) or hydrates thereof -
5 in particular lithium hydroxide monohydrate ($\text{LiOH} \cdot \text{H}_2\text{O}$). Alternatively hydrogenated fatty acids or hydrogenated hydroxy fatty acids are commonly used in the neutralization.

Suitable saponification agents are alkali metal or alkaline
10 earth metal hydroxides (or mixtures of both), in particular lithium hydroxide, or hydrates thereof. Therefore, the feed materials preferably comprise

- a) hydrogenated castor oil (HCO) and lithium hydroxide or lithium hydroxide monohydrate;
- 15 b) HCO and lithium hydroxide (or monohydrate) or calcium hydroxide;
- c) Hydrogenated castor oil fatty acid (HCOFA) and lithium hydroxide or lithium hydroxide monohydrate; and/or
- d) HCOFA and lithium hydroxide (or monohydrate) or calcium
20 hydroxide.

Suitable complexing agents are short chain fatty acids, such as acetic acid, dicarboxylic acids, such as azelaic acid, hydroxy aromatic acids such as salicylic acid, boric acid, aluminium compounds, etc. If such complexing agents are used the proportion
25 of saponification agents is usually increased with about the stoichiometric quantity of the complexing agent.

Other suitable thickening agents are clays, including coated clays, silica gels, polymers, polyureas, carbon black, dyes, etc.

Suitable lubricating oils are mineral oils, including hydro-
30 genated mineral oils, synthetic oils, such as ester oils, polyolefin oils, silicon oils, phosphate esters, polyphenyl ethers, etc. Suitable mineral lubricating oils include HVI, paraffinic MVI, naphthenic MVI and LVI oils.

To the base greases may be added additional lubricating oil and additives, such as antioxidants, anti-rust, anti-wear and extreme pressure agents, pour point depressants, metal deactivators etc.

5 Suitable laboratory-type motionless or static mixers have the form of a cylinder or tube having a length of 65.5 to 66.5 cm and a diameter of 3.3 to 3.6 cm. They are provided with an insulated heating jacket, using e.g. electric tape having a heating capacity of e.g. 400 watt per mixer, or a heating medium as the heating
10 source. The inside of the cylinder is provided with e.g. vanes, corrugated plates, perforated plates, intermeshing and intersecting bars and similar fixed mechanical obstacles causing mixing and heat exchange. A suitable motionless mixer containing perforated plates is described in e.g. British patent specification 2086249,
15 but other types may be used as well.

 Suitable mixers are: the Sulzer motionless mixer SMX type which is 655 mm long x 36 mm internal diameter and the Sulzer motionless heat exchanger SMXL type which is 665 mm long x 32.8 mm internal diameter.

20 For commercial production the sizes of the mixers require scaling up.

 According to the present process the base grease-making ingredients are preferably blended at room temperature in a container from which they are pumped through at least one motion-
25 less mixer, preferably several motionless mixers, preferably longitudinally connected. They may also be, or additionally be, connected in a parallel manner.

 The average velocity of the mixture in the mixers may be 3 kg to 50 kg/h, preferably 7 to 30 kg/h, the flow preferably being
30 laminar.

 The pressure at the entrance of the first mixer may be between 1 and 30 bar, preferably between 1 and 15 bar.

 The mixers are heated to a suitable high temperature, e.g. at least 120 °C, preferably between 120 and 250 °C, preferably up

to a maximum temperature between 180 and 230 °C, so that a grease structure is formed on cooling. Preferably the temperature of each of the heated mixers is higher than that of the preceding mixer and so on.

5 Thermocouples may be placed between the mixers for temperature control.

If desired part of the finished grease may be recycled. Also the pump at the entrance of the first mixer may be provided with a recycling circuit.

10 The subsequent removal of water can be accomplished by simply dumping the resulting grease in an open vessel, whereafter any further lubricating oil and any desired additives can be added.

Due to condensation some water may be present in the open vessel. If required complete dehydration may be accomplished using
15 conventional flash vacuum equipment.

Preferably the base grease together with additional lubricating oil and additives is pumped through a heat exchanger tube for mixing and cooling to obtain the desired consistency.

Homogenisation of the grease, if necessary, is carried out
20 using e.g. a Manton Gaulin homogeniser at 3000 psig (20.7 MPa), a mill or a rolling device etc.

The invention is illustrated by the following Examples.

EXAMPLE 1

1.3 kg feed containing:

25 12 %w hydrogenated castor oil

1.7 %w LiOH.H₂O

86.3 %w mineral lubricating oil (HVI, viscosity 10.7-11.8 cSt at 100 °C, VI = 96 (min), and 3 %w (= 39 g) water is mixed at room temperature for at least 10 minutes in a container and then pumped
30 at a flow rate of 8.3 kg/h once through a series of 4 Sulzer motionless mixers (each unit containing 2 mixing elements) which are beforehand adjusted to temperatures of 130, 170, 190 and 230 °C, respectively, by 4 200 watt electric heaters per mixer.

The finished grease is obtained at the outlet and cooled in a jacketed SULZER MONOTUBE HEAT EXCHANGER which consists of 2 units each 1540 mm long x 32.8 mm internal diameter. Each unit contains 1 SMXL type heat exchanger. The unworked/worked penetration at 25 °C was 262/273 dmm (ASTM D217-82) (1 dmm corresponds with 0.1 mm) and the dropping point was 197 °C. (Worked penetration is the penetration at 25 °C after 60 double strokes in a standard grease worker).

The first two Sulzer mixers were of the type SMX DN32, having a length of 655 mm and an internal diameter of 36 mm and the last two were of the type SMXL DN32 (heat exchanger type for boosting the heat transfer during warming up of the viscous mixture), having a length of 665 mm and an internal diameter of 32.8 mm.

Subsequently the water present in the grease was flashed off. Examples 2-6 are carried out in the arrangement of Example 1, but the flow rate is changed, resulting in different outlet temperatures, penetrations and dropping points (see Table A).

TABLE A

Example	Outlet temperature of base grease, °C	flow rate kg/h	Penetration (ASTM D217-82)		Dropping point, °C
			unmilled unworked/worked	milled unworked/worked	
			dyn	dyn	
1	218	8.3	277/287	262/273 *	197
2	204	7.4	216/313	302/319	83
3	203	8.2	247/338	319/330	194
4	205	8.9	285/334	323/330	194
5	213	9.2	359/327	289/291 *	202
6	225	8.8	334/357	332/332	198

* The worked penetration of both these greases conform to a National Lubricating Grease Institute (NLGI) 2 number i.e. 265-295.
The other greases 2, 3, 4 & 6 are NLGI 1 number i.e. 310-340 (Softer greases than 2 grade)

This table indicates that a better yield is obtained at outlet temperatures of around 213-218 °C, see especially Example 1.

C L A I M S

1. A process for the continuous manufacture of a lubricating grease comprising blending feed materials and lubricating oil and pumping the resulting blend through at least one motionless mixer which is heated to such a temperature that soap formation takes place and a resultant grease is formed.
2. A process according to claim 1, wherein the blending of feed materials and lubricating oil is carried out at room temperature.
3. A process according to claim 1 or 2, wherein the feed materials comprise
 - a) hydrogenated castor oil (HCO) and lithium hydroxide or lithium hydroxide monohydrate;
 - b) HCO and lithium hydroxide (or monohydrate) or calcium hydroxide;
 - c) Hydrogenated castor oil fatty acid (HCOFA) and lithium hydroxide or lithium hydroxide monohydrate; and/or
 - d) HCOFA and lithium hydroxide (or monohydrate) or calcium hydroxide.
4. A process according to any one of claims 1-3, wherein a series of heated motionless mixers is used, each mixer having a higher temperature than the preceding one.
5. A process according to any one of claims 1-4, wherein the motionless mixers have vanes.
6. A process according to any one of claims 1-5, wherein the mixers are heated to a temperature between 120 and 250 °C.
7. A process according to any one of claims 1-6, wherein the resulting grease is cooled in a heat exchanger tube.
8. A process according to any one of claims 1-7, wherein to the resulting grease additional lubricating oil and additives are added.
9. A process according to claim 1, substantially as hereinbefore described.
10. A lubricating grease whenever prepared, according to a process as claimed in any one of claims 1-9.