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(54) A LITHIUM HYDROXIDE COMPOSITION, A PROCESS FOR PREPARING A LITHIUM HYDROXIDE COMPOSITION, AND A PROCESS FOR USING A LITHIUM HYDROXIDE COMPOSITION

LITHIUMHYDROXIDZUSAMMENSETZUNG, VERFAHREN ZUR HERSTELLUNG EINER LITHIUMHYDROXIDZUSAMMENSETZUNG UND VERFAHREN ZUR VERWENDUNG EINER LITHIUMHYDROXIDZUSAMMENSETZUNG

COMPOSITION D'HYDROXYDE DE LITHIUM, SON PROCÉDÉ DE PRÉPARATION ET SON PROCÉDÉ D'UTILISATION

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(56) References cited:
WO-A-2004/031328
WO-A-2005/097952
WO-A-2008/058053
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EP 2 147 087 B1

Description

FIELD OF THE INVENTION

[0001] The invention relates to a lithium hydroxide composition, a process for preparing a lithium hydroxide composition, and a process for using a lithium hydroxide composition.

BACKGROUND OF THE INVENTION

[0002] Soap concentrates, for example, lithium soap concentrates, lithium complex soap concentrates, and lithium-calcium complex soap concentrates, may be utilized with a base oil, generally to thicken a base oil, to provide for a lubricating composition commonly referred to as a grease.

[0003] In addition to the individual components of a grease, another contributing factor to the final properties and characteristics of the grease is the particular process and conditions under which the soap concentrate precursors, the soap concentrate, and the grease are prepared. Process conditions such as the dispersing and mixing of the individual components and temperature variations may be significant factors affecting the soap concentrate procursors, the soap concentrate, and the resulting grease, for example, the nature of the soap crystallites and fibers formed.

[0004] For example, in situ preparation of dilute suspensions ot anhydrous lithium hydroxide monohydrate in oil may be useful for preparing soap concentrates and greases. However, the density of solid anhydrous lithium hydroxide is such that the solid tends to settle preventing the formation of a stable suspension comprising lithium hydroxide.

[0005] In addition, in the preparation of a grease, for example, a lithium grease, lithium hydroxide monohydrate is typically dissolved in a solvent such as water. The water must then be removed during the process of preparing the grease. Removing the water consumes time and energy.

[0006] U.S. Patent No. 5,236,607 to Harris et el. discloses a process for the preparation of a lithium soap thickened grease which consists of heating a mixture of an oil and a lithium base, and optionally also a calcium base, to at least 100°C., then adding a C10-C24 saturated or unsaturated fatty acid, and heating the resulting mixture at a temperature in the range of 110°C. to 200°C. until a thickened grease is obtained.

[0007] International publication number WO 2004/031328 A2 discloses a grease composition comprising a reaction product of a stable dispersion of a metal hydroxide with a number average particle size in the range 20 nanometres to 2 micrometres, a surfactant with a HLB of less than 10, a mono- and/or poly- carboxylic acid, and an oil of lubricating viscosity. The method of preparing a grease composition is also disclosed with benefits including a reduction in reaction time, amount of foam produced and environmental hazards.

[0008] WO 2005/037952 A discloses high solids content dispersions.

[0009] Despite the history of soap concentrates and greases, there is still a need for a stable lithium hydroxide composition, for example, a stable suspension comprising lithium hydroxide, and a process for preparing same, that may be utilized to provide for a soap concentrate or a grease.

SUMMARY OF THE INVENTION

[0010] The invention provides for a lithium hydroxide composition comprising lithium hydroxide according to claim 1.

[0011] The invention also provides for a process for preparing a lithium hydroxide composition according to claim 11.

[0012] The invention also provides for a process of using a lithium hydroxide composition according to claim 12.

[0013] The invention also provides for a process of using a lithium hydroxide composition according to claim 13.

DETAILED DESCRIPTION OF THE INVENTION

[0014] The invention provides for a lithium hydroxide composition that may be utilized to provide for a soap concentrate or a grease. The invention also provides for the preparing of a lithium hydroxide composition that may be utilized to provide for a soap concentrate or a grease.

[0015] The invention may provide for one or more of the following advantages.

[0016] An advantage of the invention is that a lithium hydroxide composition may be provided that is stable to separation under prolonged storage. In other words, the components of the lithium hydroxide composition do not separate or settle out, for example, during storage.

[0017] Another advantage of the invention is that a stable lithium hydroxide composition may be maintained or stored for a period of time and then used as needed in preparing a soap concentrate or a grease.

[0018] Another advantage of the invention is the flexibility that may be provided by a lithium hydroxide composition of the invention. For example, the lithium hydroxide composition may be utilized upon production to further provide for a soap concentrate or a grease, or storing or maintaining the lithium hydroxide composition for future use when needed

to provide for a soap concentrate or a grease.

[0019] Another advantage of the invention is that a reduced amount, preferably no amount, of a solvent, for example, water, is added during the preparation of a lithium hydroxide composition of the invention. A reduced amount of added solvent may provide for a lithium hydroxide composition that is more dry than a lithium hydroxide composition that is prepared from a process that utilizes an increased amount of solvent that may provide for a reduction in time and energy that is typically required for removing the solvent during, for example, the preparation of a grease.

[0020] Another advantage of the invention is that a lithium hydroxide composition may be provided that is of a suitable composition that the lithium hydroxide composition may be added to other components to provide for a soap concentrate or a grease. For example, a lithium hydroxide composition may be provided using a process of the invention at a particular location. The lithium hydroxide composition may then be relocated to another location for preparing a soap concentrate or a grease.

[0021] A lithium hydroxide composition of the invention comprises lithium hydroxide that includes any suitable form of lithium hydroxide, for example, anhydrous lithium hydroxide, crystalline lithium hydroxide monohydrate, lithium hydroxide monohydrate, or a combination thereof. An example lithium hydroxide composition of the invention comprises anhydrous lithium hydroxide and is essentially free of lithium hydroxide monohydrate. Preferably, the anhydrous lithium hydroxide is provided by dehydrating lithium hydroxide monohydrate according to a process of the invention.

[0022] A lithium hydroxide composition of the invention is generally in the form of a suspension comprising lithium hydroxide. A lithium hydroxide composition of the invention is preferably in the form of a stable suspension comprising lithium hydroxide.

[0023] The term "suspension" indicates that a lithium hydroxide composition of the invention comprises particles comprising lithium hydroxide mixed with the other components as described herein. For example, the particles comprising lithium hydroxide may be suspended in a base oil. The term "stable" indicates that a lithium hydroxide composition of the invention comprises a composition that does not readily alter its chemical composition or physical state through storage at ambient conditions. For example, the particles comprising lithium hydroxide do not readily separate out of, or settle out of, a lithium hydroxide composition of the invention. For example, the particles comprising lithium hydroxide do not readily separate out of, or settle out of, a lithium hydroxide composition of the invention stored under ambient conditions after a time period of 30 days, preferably after a time period of 60 days, and more preferably after a time period of 90 days. The term "homogenous" indicates that a lithium hydroxide composition of the invention comprises a substantially uniform structure or composition throughout a lithium hydroxide composition of the invention.

[0024] The stability of a lithium hydroxide composition of the invention provides for the ability to store a lithium hydroxide composition of the invention over long periods of time without a separating out or settling of the components of the lithium hydroxide composition. For example, a time period including from production up to 12 months may be obtained for storing a lithium hydroxide composition of the invention without a significant separating out or settling of the components of the lithium hydroxide composition. Also, for example, a lithium hydroxide composition of the invention may be used upon production as an ingredient for preparing a soap concentrate or a grease. Also, for example, a lithium hydroxide composition of the invention may be used at any time from production up to 12 months. Depending on the storage conditions for storing a lithium hydroxide composition of the invention, a storage time period of greater than 12 months may be obtained. Thus, a lithium hydroxide composition of the invention may provide for flexibility during processes of preparing a soap concentrate or a grease.

[0025] A lithium hydroxide composition of the invention should not be considered a soap concentrate because the lithium hydroxide, which is generally basic, is generally not neutralized in a lithium hydroxide composition of the invention.

[0026] A lithium hydroxide composition of the invention generally comprises lithium hydroxide in any amount that suitably provides for a lithium hydroxide composition of the invention. A lithium hydroxide composition of the invention comprises an amount of lithium hydroxide based on the total weight of the lithium hydroxide composition generally in a range of from 10 weight percent to 60 weight percent, preferably in a range of from 15 weight percent to 50 weight percent, and more preferably in a range of from 20 weight percent to 40 weight percent.

[0027] A lithium hydroxide composition of the invention generally comprises a base oil in any amount that suitably provides for a lithium hydroxide composition of the invention. A lithium hydroxide composition of the invention comprises an amount of base oil based on the total weight of the lithium hydroxide composition generally in a range of from 40 weight percent to 90 weight percent, preferably in a range of from 45 weight percent to 85 weight percent, and more preferably in a range of from 50 weight percent to 80 weight percent.

[0028] A lithium hydroxide composition of the invention generally comprises a polymer in any amount that suitably provides for a lithium hydroxide composition of the invention. A lithium hydroxide composition of the invention comprises an amount of polymer based on the total weight of the lithium hydroxide composition generally in a range of from 0.25 weight percent to 20 weight percent, preferably in a range of from 0.5 weight percent to 6 weight percent, and more preferably in a range of from 1 weight percent to 5 weight percent.

[0029] When a fatty acid component is utilized, the fatty acid component will generally be neutralized to provide for a fatty acid salt, preferably a lithium salt of hydrogenated castor oil fatty acid referred to herein as a castor oil fatty acid

lithium salt, according to a process of the invention. When a fatty acid component is utilized, a lithium hydroxide composition of the invention generally comprises a fatty acid salt in any amount that suitably provides for a lithium hydroxide composition of the invention. When a fatty acid component is utilized, a lithium hydroxide composition of the invention comprises an amount of fatty acid salt based on the total weight of the lithium hydroxide composition generally in a range of from 0.5 weight percent to 10 weight percent, preferably in a range of from 0.5 weight percent to 9 weight percent, and more preferably in a range of from 1 weight percent to 8 weight percent.

[0030] The ratio of water to lithium hydroxide of a lithium hydroxide composition of the invention may be any ratio that suitably provides for a lithium hydroxide composition of the invention. The ratio of water to lithium hydroxide is generally in a range of from 0:1 to 7:1, preferably in a range of from 0:1 to 0.75:1, and more preferably in a range of from 0:1 to 0.5:1. An advantage of the invention is that a lithium hydroxide composition may be provided without the addition of water. In other words, the ratio of water to lithium hydroxide may be 0:1. An additional advantage of the invention includes, but is not limited to, a reduced amount of water being utilized that may provide for a reduction in time and energy needed to dehydrate the resulting mixture comprising a lithium hydroxide component.

[0031] Base oils, also referred to in the art as lubricating oils, for use in providing for a lithium hydroxide composition of the invention, a soap concentrate, or a grease may typically be the same as base oils that would normally be selected for oil lubrication. The base oil may be of mineral origin, synthetic origin, vegetable origin, animal origin, or a combination thereof. Base oils of mineral origin may be mineral oils, for example, those produced by solvent refining or hydroprocessing. Base oils of synthetic origin may typically comprise mixtures of C_{10}-C_{50} hydrocarbon polymers, for example liquid polymers of alpha-olefins, ester type polymers, ether type polymers, and combinations thereof. Base oils may also include Fischer-Tropsch derived highly paraffinic products. Preferably, the base oil is of mineral origin.

[0032] Suitable examples of synthetic oils include polyolefins such an alpha-olefin oligomers and polybutene, polyalkylene glycols such as polyethylene glycol and polypropylene glycol, diesters such as di-2-ethyl hexyl sebacate and di-2-ethyl hexyl adipate, polyol esters such as trimethylolpropane ester and pentaerythritol ester, perfluorolalkyl ethers, silicone oils, polyphenyl ethers, either individually or as mixed oils.

[0033] Examples of base oils suitable for preparing a lithium hydroxide composition of the invention include medium viscosity mineral oils, high viscosity mineral oils, and combinations thereof. A base oil suitable for preparing a lithium hydroxide composition of the invention generally has a viscosity in a range of from 2 mm^2/s centistokes (cSt) at 40 °C to 600 mm^2/s (cSt) at 40 °C, preferably in a range of from 25 mm^2/s (cSt) at 40 °C to 400 mm^2/s (cSt) at 40 °C, and more preferably in a range of from 50 mm^2/s (cSt) at 40 °C to 130 mm^2/s (cSt) at 40 °C.

[0034] For example, a medium viscosity mineral oil suitable for preparing a lithium hydroxide composition of the invention may have a viscosity generally in a range of from 95 mm^2/s centistokes (cSt) at 40 °C to 115 mm^2/s (cSt) at 40 °C, preferably in a range of from 100 mm^2/s (cSt) at 40 °C to 110 mm^2/s (cSt) at 40 °C, more preferably in a range of from 105 mm^2/s (cSt) at 40 °C to 110 mm^2/s (cSt) at 40 °C, and even more preferably 108 mm^2/s (cSt) at 40 °C. Also for example, a high viscosity mineral oil suitable for preparing a lithium hydroxide composition of the invention may have a viscosity generally in a range of from 120 mm^2/s (cSt) at 40 °C to 140 mm^2/s (cSt) at 40 °C, preferably in a range of from 125 mm^2/s (cSt) at 40 °C to 135 mm^2/s (cSt) at 40 °C, more preferably in a range of from 125 mm^2/s (cSt) at 40 °C to 130 mm^2/s (cSt) at 40 °C, and even more preferably 130 mm^2/s (cSt) at 40 °C.

[0035] Suitable examples of mineral oils that may be used for preparing a lithium hydroxide composition of the invention include those having the designations "HVI" or "MVI". Suitable examples include oils having a general designation "MVI 500", "HVI 250", or "HVI 600". Polyalphaolefins and base oils of the type manufactured by the hydroisomerisation of wax may also be used.

[0036] Generally, a process of preparing a lithium hydroxide composition of the invention comprises contacting a lithium hydroxide component, a base oil, and a styrene-ethylene/propylene copolymer. The process generally comprises heating the resulting mixture to dehydrate the lithium hydroxide component to provide for a lithium hydroxide composition of the invention. Heating the resulting mixture may also dehydrate the resulting mixture comprising the lithium hydroxide component.

[0037] Examples of a suitable lithium hydroxide component for use in preparing a lithium hydroxide composition of the invention include crystalline lithium hydroxide monohydrate, lithium hydroxide monohydrate, anhydrous lithium hydroxide, and combinations thereof. When preparing a lithium hydroxide composition of the invention, the lithium hydroxide component is preferably crystalline lithium hydroxide monohydrate.

[0038] The polymer for preparing the lithium hydroxide composition of the invention is an olefin copolymers comprising styrene-ethylene/propylene copolymers, for example, a styrene-ethylene/propylene copolymer commercially available from member companies of the Shell Group having the designation "Shellvis 60".

[0039] When a fatty acid component is utilized, the fatty acid component will generally be neutralized by the lithium hydroxide component during contacting to provide for a fatty acid salt. A fatty acid salt as referred to herein comprises a salt of a fatty acid component as described herein. Preferably, a fatty acid salt is a lithium salt of hydrogenated castor oil fatty acid referred to herein as a castor oil fatty acid lithium salt.

[0040] Examples of suitable fatty acid components for preparing a lithium hydroxide composition of the invention

include fatty acids, fatty acid esters, fatty glycerides, and combinations thereof. The fatty acid component comprises carbon atoms generally in a range of from 10 carbon atoms to 24 carbon atoms (C_{10}-C_{24}), preferably in a range of from 15 carbon atoms to 18 carbon atoms (C_{15}-C_{18}). The fatty acid component may be saturated or unsaturated. Examples of suitable fatty acid components for preparing a lithium hydroxide composition of the invention include oleic acid, palmitic acid, stearic acid, and other carboxylic acids derived from tallow, hydrogenated fish oil, castor oil, wool, grease, and rosin, and combinations thereof. Examples of suitable fatty acid components for preparing a lithium hydroxide composition of the invention include hydrogenated castor oil (HCO), hydrogenated castor oil fatty acid (HCOFA), and combinations thereof, preferably hydrogenated castor oil fatty acid (HCOFA). Hydrogenated castor oil (HCO) is the glyceride of 12-hydroxystearic acid. 12-hydroxystearic acid is a preferred fatty acid.

[0041] Hydrogenated castor oil fatty acid (referred to herein as HCOFA) generally comprises at least 85 weight percent of 12-hydroxystearic acid based on the total weight of HCOFA. HCOFA may comprise minor amounts of additional components. Examples of additional components include palmitic acid (C16), stearic acid (C18), arachidic acid (C20), 12-ketostearic acid, and combinations thereof. As used herein, the term "hydrogenated castor oil fatty acid" ("HCOFA") refers to a composition comprising an amount of 12-hydroxystearic acid that provides for a process of the invention, generally an amount comprising at least 85 weight percent 12-hydroxystearic acid based on the total weight of HCOFA, preferably an amount comprising in a range of from 85 to 87 weight percent 12-hydroxystearic acid based on the total weight of HCOFA. An example HCOFA that may be used for preparing a lithium hydroxide composition of the invention includes CENWAX A from Arizona Chemical, Jacksonville, Florida, USA.

[0042] Contacting of the components to provide for a lithium hydroxide composition of the invention generally refers to a contacting that is conducted at a temperature, a pressure, and a time period that suitably provides for a lithium hydroxide composition of the invention. Contacting generally comprises a heating of the lithium hydroxide component, preferably lithium hydroxide monohydrate, more preferably crystalline lithium hydroxide monohydrate, that provides for a dehydrating of the lithium hydroxide component to provide for a lithium hydroxide composition of the invention. Heating may also provide for a dehydrating of the resulting mixture comprising the lithium hydroxide component. For example, a process of the invention comprising a heating and a dehydrating of a lithium hydroxide component may also provide for a heating and a dehydrating of the mixture comprising the lithium hydroxide component.

[0043] Dehydrating may provide for a reduction in time and energy typically required to remove a solvent, for example, water, utilized during the preparation of a soap concentrate or a grease. When a fatty acid component is utilized in preparing a lithium hydroxide composition of the invention, contacting generally provides for a neutralizing of the fatty acid component with the lithium hydroxide component to provide for a fatty acid salt, for example, a castor oil fatty acid lithium salt.

[0044] Contacting of the components may be conducted in any order that suitably provides for a lithium hydroxide composition of the invention. Contacting of the components may be conducted using any means that suitably provide for a lithium hydroxide composition of the invention. Examples of suitable contacting means include mixing, stirring, circulating, and combinations thereof.

[0045] The temperature during contacting may be any temperature that suitably provides for a heating and a dehydrating of a lithium hydroxide component, a resulting mixture comprising a lithium hydroxide component, or a combination thereof, to provide for a lithium hydroxide composition of the invention and is generally a temperature found in batch processing techniques. Generally, the temperature is in a range of from 80 °C to 280 °C, preferably in a range of from 85 °C to 225 °C, and more preferably in a range of from 90 °C to 215 °C.

[0046] The pressure during contacting may be any pressure that suitably provides for a heating and a dehydrating of a lithium hydroxide component, a resulting mixture comprising a lithium hydroxide component, or a combination thereof, to provide for a lithium hydroxide composition of the invention and is generally a pressure found in batch processing techniques. Generally, the pressure is in a range of from atmospheric, in other words, 0 kilopascal (kPa), to 1380 kPa, preferably in a range of from 0 kPa to 690 kPa, and more preferably in a range of from 0 kPa to 345 kPa.

[0047] The time period during contacting may be any time period that suitably provides for a heating and a dehydrating of a lithium hydroxide component, a resulting mixture comprising a lithium hydroxide component, or a combination thereof, to provide for a lithium hydroxide composition of the invention and is generally a time period found in batch processing techniques. Generally, the time period is in a range of from 0.5 hours to 8 hours, preferably in a range of from 1 hour to 6 hours, and more preferably in a range of from 1 hour to 3 hours.

[0048] A process of preparing a lithium hydroxide composition of the invention may comprise the use of an anti-foaming agent. Examples of suitable anti-foaming agents include any anti-foaming agent typically used in the art of preparing a soap concentrate or a grease. Examples of suitable anti-foaming agents for preparing a lithium hydroxide composition of the invention include an anti-foaming agent commercially available from Rhein Chemie under the designation "PC1644".

[0049] Some of the polymers, for example, a styrene-ethylene/propylene copolymer commercially available from member companies of the Shell Group having the designation "Shellvis 60", that may be utilized in a process of the invention may help reduce foaming during dehydrating of a mixture comprising a lithium hydroxide component. Reducing foaming

may provide for a reduced time period, in other words, less time being needed, during contacting of the components to provide for a lithium hydroxide composition of the invention.

[0050] The lithium hydroxide component, preferably lithium hydroxide monohydrate, more preferably crystalline lithium hydroxide monohydrate, that is initially present during a process of the invention is dehydrated so that anhydrous lithium hydroxide, also referred to as dehydrated lithium hydroxide, is present in a lithium hydroxide composition of the invention. The amount of lithium hydroxide component that is dehydrated to provide for the anhydrous lithium hydroxide based on the total amount of lithium hydroxide component initially present is generally in a range of from 80 percent to 100 percent, preferably in a range of from 90 percent to 100 percent, and more preferably in a range of from 95 percent to 100 percent. For example, if an amount of lithium hydroxide monohydrate that is dehydrated is 100 percent, then a lithium hydroxide composition of the invention will generally comprise anhydrous lithium hydroxide and generally no lithium hydroxide monohydrate. Also for example, if an amount of lithium hydroxide monohydrate that is dehydrated is 80 percent, then a lithium hydroxide composition of the invention will comprise anhydrous lithium hydroxide and lithium hydroxide monohydrate.

[0051] Utilizing a process of the invention at different locations may provide for the flexibility to provide for a lithium hydroxide composition of the invention at one location and then send the lithium hydroxide composition to another location to provide for a soap concentrate or a grease depending on the needs of the sent-to location.

[0052] For example, a lithium hydroxide composition of the invention may be prepared utilizing batch processing. Also for example, a lithium hydroxide composition of the invention may be utilized to provide for a soap concentrate or a grease utilizing batch processing, conventional reconstitution techniques, extruder techniques, or a combination thereof. Fo: example, a lithium hydroxide composition of the invention may be prepared utilizing batch processing to provide for a resulting lithium hydroxide composition of the invention that may then be utilized to provide for a soap concentrate or a grease utilizing batch processing, conventional reconstitution techniques, extruder techniques, or a combination thereof.

[0053] Batch processing as referred to herein generally comprises the use of one or more large kettles that may be equipped with, for example, paddle agitation, stirring, heating, external recirculation systems capable of pumping the contents from the bottom of the kettle to the top, and combinations thereof. Any size kettle that suitably provides for a lithium hydroxide composition of the invention, a soap concentrate, or a grease may be utilized. While the examples disclosed herein utilized a smaller size kettle of the kind that is generally used in a laboratory setting, a process of the present invention may be utilized in larger commercial size kettles that may be of a size generally in a range of from 1,000 liters to 20,000 liters, preferably in a range of from 2,000 liters to 15,000 liters, and more preferably in a range of from 3,000 liters to 10,000 liters. Examples of suitable kettles include open kettles and pressurized kettles. An example grease kettle is equipped with stirring, heating, and an external recirculation system, capable of pumping the contents from the bottom of the kettle to the top.

[0054] The reconstitution to provide for a soap concentrate or a grease may take place at the same location(s) as the preparing of a lithium hydroxide composition of the invention or at a different location(s) than the preparing of a lithium hydroxide composition of the invention.

[0055] Storing a lithium hydroxide composition of the invention may be conducted utilizing any temperature and technique used in the art of storing a soap concentrate or a grease. Examples of storing include the use of drums, pails, totes, and combinations thereof, preferably with caustic resistant liners. Storage life may be further increased by storing a lithium hydroxide composition of the invention under an inert atmosphere, for example, nitrogen or argon. A lithium hydroxide composition of the invention is generally allowed to cool prior to storing.

[0056] The temperature of storing a lithium hydroxide composition of the invention may be any temperature that suitably provides for storage. Generally, the temperature is in a range of from 10 °C to 40 °C, preferably in a range of from 15 °C to 35 °C, and more preferably in a range of from 20 °C to 30 °C.

[0057] A soap concentrate prepared utilizing a lithium hydroxide composition of the invention comprises a weight percent of lithium hydroxide composition based on the total weight of soap concentrate generally in a range of from 1 weight percent to 20 weight percent, preferably in a range of from 5 weight percent to 20 weight percent, and more preferably in a range of from 8 weight percent to 15 weight percent.

[0058] Soap concentrates prepared utilizing a lithium hydroxide composition of the invention include simple soap concentrates, complex soap concentrates, and combinations thereof. Complex soap concentrates include simple soap concentrates additionally comprising a complexing agent.

[0059] Examples of suitable soap concentrates that may be prepared utilizing a lithium hydroxide composition of the invention include lithium soap concentrates, lithium complex soap concentrates, lithium-calcium soap concentrates, and combinations thereof. Examples of preferred soap concentrates prepared utilizing a lithium hydroxide composition of the invention include lithium soap concentrates and lithium complex soap concentrates.

[0060] A soap concentrate, for example a lithium soap concentrate, may be prepared by contacting a lithium hydroxide composition of the invention with a fatty acid component as referred to herein. The resulting soap concentrate may then be contacted with a base oil to provide for a grease. Also, for example, a lithium hydroxide composition of the invention may be contacted with a fatty acid component and a base oil to provide for a soap concentrate in situ during the providing

of the resulting grease.

[0061] Lithium complex soap concentrates are soap concentrates wherein a complexing agent has been incorporated into a lithium soap concentrate. Examples of suitable complexing agents include dibasic acids, salts thereof, and combinations thereof, for example azelaic acid, boric acid, lithium borate, and combinations thereof, preferably boric acid.

[0062] A grease prepared utilizing a lithium hydroxide composition of the invention comprises a weight percent of lithium hydroxide composition based on the total weight of grease generally in a range of from 1 weight percent to 20 weight percent, preferably in a range of from 2 weight percent to 15 weight percent, and more preferably in a range of from 2 weight percent to 12 weight percent.

[0063] Examples of greases that may be prepared utilizing a lithium hydroxide composition of the invention include lithium greases, lithium complex greases, lithium-calcium complex greases, and combinations thereof. Examples of preferred greases that may be prepared utilizing a lithium hydroxide composition of the invention include lithium greases and lithium complex greases.

[0064] A grease prepared utilizing a lithium hydroxide composition of the invention may comprise additional additives, in amounts normally used in this field of application, to impart certain desirable characteristics to the grease including oxidation stability, tackiness, extreme pressure properties, corrosion inhibition, reduced friction and wear, and combinations thereof.

[0065] Examples of suitable additional additives include antioxidants, anti-rust additives, anti-wear and extreme pressure additives, pour point depressants, metal deactivators, and combinations thereof.

[0066] A grease prepared utilizing a lithium hydroxide composition of the invention may comprise from 0.1 weight percent to 10 weight percent, preferably from 0.1 weight percent to 5 weight percent, more preferably from 0.1 weight percent to 2 weight percent, and even more preferably from 0.2 weight percent to 1 weight percent of one or more additional additives as referred to herein based on the total weight of the grease. For example, a combination of additional additives may be needed to achieve a higher weight percent of additional additive, for example 10 weight percent.

[0067] Examples of suitable additional additives include zinc salts such as zinc dialkyl or diaryl dithiophosphates, borates, molybdenum dithiophosphate, substituted thiadiazoles, polymeric nitrogen/phosphorus compounds made, for example, by reacting a dialkoxy amine with a substituted organic phosphate, amine phosphates, sulphurised sperm oils of natural or synthetic origin, sulphurised lard, sulphurised esters, sulphurised fatty acid esters, sulphurised materials, organophosphates, for example according to the formula $(OR)_3P=O$ where R is an alkyl, aryl or aralkyl group, and triphenyl phosphorothionate; one or more overbased metal-containing detergents, such as calcium or magnesium alkyl salicylates, alkylarylsulphonates or alkylsulphonates; ashless dispersant additives, such as reaction products of polyisobutenyl succinic anhydride and an amine or ester; antioxidants, such as hindered phenols or amines, for example phenyl alpha naphthylamine; antirust additives such as zinc naphthenate; friction-modifying additives; viscosity-index improving agents; pour point depressing additives; tackiness agents, and combinations thereof. Solid materials such as graphite, finely divided molybdenum disulphide, talc, metal powders, and various polymers such as polyethylene wax may also be added to impart special properties.

[0068] Where the term aryl is referred to herein, the aryl group is preferably a phenyl group or a naphthyl group. Where the term aralkyl is referred to herein, the aralkyl group is preferably a benzyl group or methyl naphthenate.

[0069] A grease prepared utilizing a lithium hydroxide composition of the invention may comprise an additional additive comprising a single zinc dithiophosphate or a combination of two or more zinc dithiophosphates. A grease prepared utilizing a lithium hydroxide composition of the invention may comprise an additional additive comprising a single ashless dithiocarbamate or a combination of two or more ashless dithiocarbamates.

EXAMPLES

EXAMPLE 1 (Comparative)

[0070] A grease kettle equipped with stirring, gas-fired heating, and an external recirculation system, capable of pumping the contents from the bottom of the kettle to the top, was charged with 4999.5 grams of high viscosity index (HVI) mineral oil having a viscosity of 50 mm^2/s (cSt) at 40 °C (104 °F) (generally having a designation "HVI 250"), 4999.5 grams of lithium hydroxide monohydrate, and 1 gram of antifoam agent (obtained from Rhein Chemie under the designation "PC1644"). The resulting mixture was stirred and heated to above 212 °F (100 °C) at a heating rate of 4.4 °F (2.444 °C) per minute. At 217 °F (102.8 °C), foaming was observed. A few drops of the PC1644 antifoam agent were added. The foam decreased for a few minutes and then the resulting mixture started to foam again. After four hours, the foaming stopped. After the foaming stopped, the kettle was heated to 300 °F (148.9 °C). The temperature of the contents of the gas-fired kettle was maintained at 300 °F (148.9 °C) for 20 minutes while the contents of the kettle were stirred and circulated from bottom to top using a gear pump. After 20 minutes, heating of the kettle contents was stopped and the kettle was stirred for two hours and allowed to cool to room temperature (70 °F to 80 °F) (21.11 °C to 26.67 °C). The resulting lithium hydroxide composition, after dehydration of the lithium hydroxide monohydrate, comprised 36.36

weight percent dehydrated lithium hydroxide, 63.63 weight percent HVI 250 base oil, and 0.01 weight percent PC1644 antifoam agent. The resulting lithium hydroxide composition was then transferred to a plastic five-gallon pail. After storage for 24 days at room temperature (70 °F to 80 °F) (21.11 °C to 26.67 °C), the top half of the container contained base oil and the dehydrated lithium hydroxide had settled to the bottom of the pail. After storage for 5 months at room temperature (70 °F to 80 °F) (21.11 °C to 26.67 °C), the appearance was generally the same as after the first 24 days.

EXAMPLE 2 (Inventive)

[0071] A grease kettle as described herein in EXAMPLE 1 was charged with 4600 grams of high viscosity index (HVI) mineral oil having a viscosity of 50 mm^2/s (cSt) at 40 °C (104 °F) (generally having a designation "HVI 250"), 5000 grams of lithium hydroxide monohydrate, 300 grams of a styrene-ethylene/propylene copolymer (obtained from member companies of the Shell Group under the designation "Shellvis 60"), and 100 grams of hydrogenated castor oil fatty acid (HCOFA). The resulting mixture was stirred and heated to 223.5 °F (106.4 °C) at a heating rate of 3 °F (1.667 °C) per minute. The temperature was maintained at 223.5 °F (106.4 °C) for 75 minutes to dehydrate the mixture (neutralize the hydrogenated castor oil fatty acid with lithium hydroxide and distill off the resulting water). No foaming was observed during the dehydration of the mixture. The temperature of the kettle contents was then increased to 300 °F (148.9 °C) over a time period of 60 minutes. The temperature was then increased to 400 °F (204.4 °C) over a time period of 60 minutes with no observable change in the resulting composition. The resulting composition was circulated for 20 minutes and allowed to cool overnight. The resulting lithium hydroxide composition, after dehydration of the lithium hydroxide monohydrate and neutralization of the HCOFA, comprised 58.53 weight percent HVI 250 base oil, 36.35 weight percent dehydrated lithium hydroxide, 1.30 weight percent castor oil fatty acid lithium salt, and 3.82 weight percent Shellvis 60 polymer. The resulting lithium hydroxide composition was then transferred to a five-gallon pail. After storage for 5 months at room temperature (70 °F to 80 °F) (21.11 °C to 26.67 °C), it was observed that the resulting lithium hydroxide composition in the pail had not separated and was of a similar appearance and consistency as when the resulting lithium hydroxide composition was initially placed in the pail.

EXAMPLE 3 (Inventive)

[0072] A grease kettle as described herein in EXAMPLE 1 was charged with 600 grams of a styrene-ethylene/propylene copolymer commercially available from member companies of the Shell Group under the designation "Shellvis 60" and 9400 grams of high viscosity index (HVI) mineral oil having a viscosity of 50 mm^2/s (cSt) at 40 °C (104 °F) (generally having a designation "HVI 250"). The resulting mixture was stirred and heated to 300 °F (148.9 °C) at a heating rate of 3 °F (1.667 °C) per minute. The resulting composition was circulated for 20 minutes and then was allowed to cool with stirring to a temperature below 212 °F (100 °C). A resulting viscous six percent (6%) concentrate of Shellvis 60 in HVI 250 was obtained and stored overnight.

[0073] A 5000 gram quantity of the resulting six percent (6%) concentrate of Shellvis 60 in HVI 250 and 5000 grams of lithium hydroxide monohydrate were then added to the empty grease kettle and heated to 300 °F (148.9 °C) with stirring. After circulating for 20 minutes, the heat to the kettle was stopped. At 230 °F (110 °C), the resulting lithium hydroxide composition was pumped to a pail and the resulting lithium hydroxide composition appeared to be thick and smooth. The resulting lithium hydroxide composition, after dehydration of the lithium hydroxide monohydrate, comprised 59.82 weight percent HVI 250 base oil, 36.36 weight percent dehydrated lithium hydroxide, and 3.82 weight percent Shellvis 60. After storing for 12 months at room temperature (70 °F to 80 °F) (21.11 °C to 26.67 °C), it was observed that the resulting lithium hydroxide composition in the pail had not separated and appeared to be of a similar appearance and consistency as when the resulting lithium hydroxide composition was initially placed in the pail.

[0074] TABLE 1 discloses a summary of various information from EXAMPLES 1-3.

TABLE 1

EXAMPLE	Components (weight percent)	Initial Appearance	Storage Time (approximate)	Appearance After Storage Time
1*	36.36 wt.% LiOH; 63.63 wt.% HVI base oil; 0.01 wt.% PC1644 antifoam agent	Semi-fluid homogeneous mixture	(a) 24 days; and (b) 5 months	After (a) and (b): Major separation; most of the LiOH had settled to the bottom of the pail with a significant amount of oil on top

(continued)

EXAMPLE	Components (weight percent)	Initial Appearance	Storage Time (approximate)	Appearance After Storage Time
2**	36.35 wt.% LiOH; 58.53 wt.% HVI base oil; 1.3 wt.% castor oil fatty acid lithium salt; and 3.82 wt.% Shellvis 60 polymer	Stable, homogenous suspension	5 months	No separation of the components; initial appearance and appearance after storing were generally the same
3**	36.36 wt.% LiOH; 59.82 wt.% HVI base oil; and 3.82 wt.% Shellvis 60 polymer	Stable, homogenous suspension	12 months	No separation of the components; initial appearance and appearance after storing were generally the same
* - Comparative ** - Inventive				

Claims

1. A lithium hydroxide composition comprising lithium hydroxide, a base oil, and an olefin copolymer comprising a styrene-ethylene/propylene copolymer.

2. The lithium hydroxide composition according to claim 1 wherein the lithium hydroxide is present in an amount in a range of from 10 weight percent to 60 weight percent based on the total weight of the composition.

3. The lithium hydroxide composition according to claim 1 or claim 2 wherein the base oil is selected from the group consisting of medium viscosity mineral oils having viscosity in a range of from 95 mm^2/s centistokes (cSt) at 40 °C to 115 mm^2/s (cSt) at 40 °C, high viscosity mineral oils having viscosity in a range of from 120 mm^2/s (cSt) at 40 °C to 140 mm^2/s (cSt) at 40 °C, and combinations thereof.

4. The lithium hydroxide composition according to any previous claim wherein the base oil is present in an amount in a range of from 40 weight percent to 90 weight percent based on the total weight of the composition.

5. The lithium hydroxide composition according to any previous claim wherein the olefin copolymer is present in an amount in a range of from 0.25 weight percent to 20 weight percent based on the total weight of the composition.

6. The lithium hydroxide composition according to any previous claim wherein the composition further comprises a fatty acid salt and further wherein the fatty acid salt is present in an amount in a range of from 0.5 weight percent to 10 weight percent based on the total weight of the composition.

7. The lithium hydroxide composition according to claim 6 wherein the fatty acid salt comprises a salt of a fatty acid component selected from the group consisting of fatty acids, fatty acid esters, fatty glycerides, and combinations thereof.

8. The lithium hydroxide composition according to any previous claim wherein the composition is in the form of a suspension.

9. The lithium hydroxide composition according to any previous claim wherein the composition is in the form of a stable suspension.

10. The lithium hydroxide composition according to any previous claim prepared by a process comprising contacting a lithium hydroxide component, a base oil, and an olefin copolymer comprising a styrene-ethylene/propylene copolymer.

11. A process for preparing a lithium hydroxide composition comprising contacting a lithium hydroxide component, a base oil, and an olefin copolymer comprising a styrene-ethylene/propylene copolymer.

12. A process of using a lithium hydroxide composition for preparing a soap concentrate comprising contacting the lithium hydroxide composition with a fatty acid component wherein the lithium hydroxide composition comprises lithium hydroxide, a base oil, and an olefin copolymer comprising a styrene-ethylene/propylene copolymer.

13. A process of using a lithium hydroxide composition for preparing a grease comprising contacting the lithium hydroxide composition with a fatty acid component and a base oil wherein the lithium hydroxide composition comprises lithium hydroxide, a base oil, and an olefin copolymer comprising a styrene-ethylene/propylene copolymer.

Patentansprüche

1. Lithiumhydroxidzusammensetzung, umfassend Lithiumhydroxid, ein Grundöl und ein Olefincopolymer, das ein Styrol-Ethylen/Propylen-Copolymer umfasst.

2. Lithiumhydroxidzusammensetzung nach Anspruch 1, wobei das Lithiumhydroxid in einer Menge in einem Bereich von 10 Gewichtsprozent bis 60 Gewichtsprozent, bezogen auf das Gesamtgewicht der Zusammensetzung, vorliegt.

3. Lithiumhydroxidzusammensetzung nach Anspruch 1 oder 2, wobei das Grundöl aus der Gruppe bestehend aus mittelviskosen Mineralölen mit einer Viskosität in einem Bereich von 95 mm^2/s Zentistokes (cSt) bei 40°C bis 115 mm^2/s (cSt) bei 40°C, hochviskosen Mineralölen mit einer Viskosität in einem Bereich von 120 mm^2/s (cSt) bei 40°C bis 140 mm^2/s (cSt) bei 40°C und Kombinationen davon ausgewählt ist.

4. Lithiumhydroxidzusammensetzung nach einem der vorhergehenden Ansprüche, wobei das Grundöl in einer Menge in einem Bereich von 40 Gewichtsprozent bis 90 Gewichtsprozent, bezogen auf das Gesamtgewicht der Zusammensetzung, vorliegt.

5. Lithiumhydroxidzusammensetzung nach einem der vorhergehenden Ansprüche, wobei das Olefincopolymer in einer Menge in einem Bereich von 0,25 Gewichtsprozent bis 20 Gewichtsprozent, bezogen auf das Gesamtgewicht der Zusammensetzung, vorliegt.

6. Lithiumhydroxidzusammensetzung nach einem der vorhergehenden Ansprüche, wobei die Zusammensetzung ferner ein Fettsäuresalz umfasst und ferner das Fettsäuresalz in einer Menge in einem Bereich von 0,5 Gewichtsprozent bis 10 Gewichtsprozent, bezogen auf das Gesamtgewicht der Zusammensetzung, vorliegt.

7. Lithiumhydroxidzusammensetzung nach Anspruch 6, wobei das Fettsäuresalz ein Salz einer Fettsäurekomponente aus der Gruppe bestehend aus Fettsäuren, Fettsäureestern, Fettglyceriden und Kombinationen davon umfasst.

8. Lithiumhydroxidzusammensetzung nach einem der vorhergehenden Ansprüche, wobei die Zusammensetzung in Form einer Suspension vorliegt.

9. Lithiumhydroxidzusammensetzung nach einem der vorhergehenden Ansprüche, wobei die Zusammensetzung in Form einer stabilen Suspension vorliegt.

10. Lithiumhydroxidzusammensetzung nach einem der vorhergehenden Ansprüche, hergestellt durch ein Verfahren, bei dem man eine Lithiumhydroxidkomponente, ein Grundöl und ein Olefincopolymer, das ein Styrol-Ethylen/Propylen-Copolymer umfasst, in Berührung bringt.

11. Verfahren zur Herstellung einer Lithiumhydroxidzusammensetzung, bei dem man eine Lithiumhydroxidkomponente, ein Grundöl und ein Olefincopolymer, das ein Styrol-Ethylen/Propylen-Copolymer umfasst, in Berührung bringt.

12. Verfahren zur Verwendung einer Lithiumhydroxidzusammensetzung zur Herstellung eines Seifenkonzentrats, wobei man die Lithiumhydroxidzusammensetzung mit einer Fettsäurekomponente in Berührung bringt, wobei die Lithiumhydroxidzusammensetzung Lithiumhydroxid, ein Grundöl und ein Olefincopolymer, das ein Styrol-Ethylen/Propylen-Copolymer umfasst, umfasst.

13. Verfahren zur Verwendung einer Lithiumhydroxidzusammensetzung zur Herstellung eines Schmierfetts, wobei man die Lithiumhydroxidzusammensetzung mit einer Fettsäurekomponente und einem Grundöl in Berührung bringt, wobei die Lithiumhydroxidzusammensetzung Lithiumhydroxid, ein Grundöl und ein Olefincopolymer, das ein Styrol-

Ethylen/Propylen-Copolymer umfasst, umfasst.

Revendications

1. Composition d'hydroxyde de lithium comprenant de l'hydroxyde de lithium, une huile de base, et un copolymère oléfinique comprenant un copolymère styrène-éthylène/propylène.

2. Composition d'hydroxyde de lithium selon la revendication 1 dans laquelle l'hydroxyde de lithium est présent dans une quantité dans une gamme de 10 pour cent en poids à 60 pour cent en poids, rapporté au poids total de la composition.

3. Composition d'hydroxyde de lithium selon la revendication 1 ou la revendication 2 dans laquelle l'huile de base est choisie dans le groupe constitué par les huiles minérales de viscosité moyenne ayant une viscosité dans une gamme de 95 mm^2/s ou centistokes (cSt) à 40 °C à 115 mm^2/s (cSt) à 40 °C, les huiles minérales de viscosité élevée ayant une viscosité dans une gamme de 120 mm^2/s (cSt) à 40 °C à 140 mm^2/s (cSt) à 40 °C, et les combinaisons de celles-ci.

4. Composition d'hydroxyde de lithium selon une quelconque revendication précédente dans laquelle l'huile de base est présente dans une quantité dans une gamme de 40 pour cent en poids à 90 pour cent en poids, rapporté au poids total de la composition.

5. Composition d'hydroxyde de lithium selon une quelconque revendication précédente dans laquelle le copolymère oléfinique est présent dans une quantité dans une gamme de 0,25 pour cent en poids à 20 pour cent en poids, rapporté au poids total de la composition.

6. Composition d'hydroxyde de lithium selon une quelconque revendication précédente, la composition comprenant en outre un sel d'acide gras et dans laquelle, en outre, le sel d'acide gras est présent dans une quantité dans une gamme de 0,5 pour cent en poids à 10 pour cent en poids, rapporté au poids total de la composition.

7. Composition d'hydroxyde de lithium selon la revendication 6 dans laquelle le sel d'acide gras comprend un sel d'un composant d'acide gras choisi dans le groupe constitué par les acides gras, les esters d'acides gras, les glycérides gras, et les combinaisons de ceux-ci.

8. Composition d'hydroxyde de lithium selon une quelconque revendication précédente, la composition se présentant sous la forme d'une suspension.

9. Composition d'hydroxyde de lithium selon une quelconque revendication précédente, la composition se présentant sous la forme d'une suspension stable.

10. Composition d'hydroxyde de lithium selon une quelconque revendication précédente préparée par un procédé comprenant la mise en contact d'un composant d'hydroxyde de lithium, d'une huile de base, et d'un copolymère oléfinique comprenant un copolymère styrène-éthylène/propylène.

11. Procédé de préparation d'une composition d'hydroxyde de lithium comprenant la mise en contact d'un composant d'hydroxyde de lithium, d'une huile de base, et d'un copolymère oléfinique comprenant un copolymère styrène-éthylène/propylène.

12. Procédé d'utilisation d'une composition d'hydroxyde de lithium pour la préparation d'un concentré de savon comprenant la mise en contact de la composition d'hydroxyde de lithium avec un composant d'acide gras, la composition d'hydroxyde de lithium comprenant de l'hydroxyde de lithium, une huile de base, et un copolymère oléfinique comprenant un copolymère styrène-éthylène/propylène.

13. Procédé d'utilisation d'une composition d'hydroxyde de lithium pour la préparation d'une graisse comprenant la mise en contact de la composition d'hydroxyde de lithium avec un composant d'acide gras et une huile de base, la composition d'hydroxyde de lithium comprenant de l'hydroxyde de lithium, une huile de base, et un copolymère oléfinique comprenant un copolymère styrène-éthylène/propylène.

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- US 5236607 A, Harris **[0006]**
- WO 2004031328 A2 **[0007]**
- WO 2005037952 A **[0008]**