



(11)

EP 4 139 426 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention of the grant of the patent:

21.01.2026 Bulletin 2026/04

(21) Application number: **21721618.3**

(22) Date of filing: **20.04.2021**

(51) International Patent Classification (IPC):

C10M 169/02 ^(2006.01) **C10N 20/02** ^(2006.01)
C10N 20/00 ^(2006.01) **C10N 30/06** ^(2006.01)
C10N 30/10 ^(2006.01) **C10N 30/00** ^(2006.01)
C10N 40/02 ^(2006.01) **C10N 50/10** ^(2006.01)

(52) Cooperative Patent Classification (CPC):
(C-Sets available)

C10M 169/02; C10M 2201/1036; C10M 2203/0206;
C10M 2203/022; C10M 2205/0285;
C10M 2207/1285; C10M 2217/0456;
C10M 2219/0466; C10M 2223/045; C10N 2020/02;
C10N 2020/065; C10N 2020/071; C10N 2030/06;
C10N 2030/10; C10N 2030/26; (Cont.)

(86) International application number:

PCT/IB2021/053227

(87) International publication number:

WO 2021/214641 (28.10.2021 Gazette 2021/43)

(54) **HIGH PERFORMANCE GREASE COMPOSITIONS WITH A RENEWABLE BASE OIL**

HOCHLEISTUNGSFETTZUSAMMENSETZUNGEN MIT ERNEUERBAREM BASISÖL

COMPOSITIONS DE GRAISSE À HAUTE PERFORMANCE AVEC UNE HUILE DE BASE
RENOUVELABLE

(84) Designated Contracting States:

**AL AT BE BG CH CY CZ DE DK EE ES FI FR GB
GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO
PL PT RO RS SE SI SK SM TR**

(30) Priority: **22.04.2020 US 202063013578 P**

(43) Date of publication of application:
01.03.2023 Bulletin 2023/09

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(52) Cooperative Patent Classification (CPC): (Cont.)
C10N 2030/40; C10N 2030/64; C10N 2030/68;
C10N 2030/74; C10N 2030/76; C10N 2040/02;
C10N 2050/10

C-Sets

C10M 2203/0206, C10N 2020/02, C10N 2020/065,
C10N 2020/071;

C10M 2207/1285, C10N 2010/02;
C10M 2219/0466, C10N 2010/04;
C10M 2223/045, C10N 2010/04

Description

FIELD OF THE INVENTION

[0001] This invention relates to the development of novel grease compositions and processes thereof based on different thickeners in renewable base oils for the first time. The superior performance characteristics of these novel grease compositions with and without performance additives has been compared with the greases prepared identically in conventional mineral and synthetic base oils.

BACKGROUND OF THE INVENTION

[0002] Overbased calcium sulfonates were reported in 1940's primarily as additives in motor oils for corrosion and oxidation protection. Overbased calcium sulfonates (OBCS) as thickener for greases were reported in 1960's and were essentially prepared in conventional mineral and or synthetic base oils like PAO's. These OBCS greases require over > 50 % thickener content to get desired consistency, thus lacks in sufficient base oil needed for lubrication. Such reported greases possess low drop point thus limited high temperature application capabilities, poor cold climate flowability and stability issues. Therefore, the properties of these fully formulated greases were inferior to then popular lithium complex greases and also significantly more expensive and thus did not get considerable attention.

[0003] In 1985 Muir et al (US No 4,560, 489) disclosed novel process and composition for making overbased calcium sulfonate complex (OBCSC) based grease containing colloiddally dispersed calcium carbonate in the form of crystalline calcite, calcium borate and calcium soap of soap-forming aliphatic monocarboxylic acid containing atleast 12 carbon atoms exemplified as 12-hydroxystearic acid. The grease compositions disclosed in this invention were prepared either in mineral or in synthetic base oils derived from mineral oil e.g., PAO. The unique composition and process disclosed in this invention resulted in lower thickener content by virtue of complexing, high drop point, extreme pressure, water resistant and rust preventive properties. This triggered the widespread applications of these (OBCSC) greases in the industry specially in high temperature, heavy loads and water borne applications. The performance characteristics of this class of OBCSC greases reported to be superior to most popular lithium complex greases.

[0004] In recent past overbased calcium sulfonate base greases have witnessed exponential growth in wide range of application areas (NLGI production survey). The potential reason for the growth of these greases are attributed due to the uncertainties in the supply of lithium, an essential component for making lithium greases due to their ever-increasing use in lithium batteries in electronics and for electric vehicles. The increasing cost of lithium has reduced the price parity between lithium complex and calcium sulfonate greases, leading to increasing interest in calcium sulfonate greases. Some recent publications and articles indicate that calcium sulfonate greases could be potential alternate to lithium greases.

[0005] There is ever increasing demand of using environment friendly lubricants and greases in environment sensitive applications like, mining, agriculture, railways, manufacturing and marines etc. Different countries have different labeling protocols and legislative compliance to promote biodegradable or renewable oil-based lubricants. In marine applications, using Vessel General Permit (VGP) complaint greases is likely to be mandated in near future. The lubricating greases based on vegetable and their synthetic esters are well reported. The thickeners used to formulate this class of environment friendly / biodegradable greases are primarily based on lithium, calcium, aluminum complex and clay-based thickeners. The major challenge in the growth of these biodegradable greases is their inherent inferior performance compared to corresponding mineral or synthetic oil like PAO's based greases.

[0006] On the other hand, efforts to formulate high performance biodegradable / renewable oil based overbased calcium sulfonate grease comparable to mineral or synthetic PAO based greases have not been very successful so far. One of the challenges to the formulators is that during gelation / conversion step and at elevated temperatures, unlike mineral oil / synthetic PAO's, newtonian overbased calcium sulfonate tend to react by itself with vegetable oils or esters derived from such oils due to its polarity and composition. This inhibits the conversion of amorphous calcium carbonate to its crystalline calcite form, an essential step in gelation process. Whereas, it is highly desirable to develop environment friendly overbased calcium sulfonate grease comparable to mineral or synthetic based oil due its certain expected inherent properties i.e., high temperature, extreme pressure & anti-wear, water resistance and rust protection.

[0007] The high performances characteristics of a grease, in general, are characterized by its capability to perform at high temperatures (> 250 °F), under heavy load conditions, humid environment, protect equipment from rust and corrosion. The patents and reports as described in following embodiments have indicated very limited scope of those greases to be true high-performance multi-purpose. The calcium sulfonate greases reported so far in following claims are either semi-fluid type soft grease, not for bearing application or has been assisted to thicken to desired consistency with the help of other supporting thickeners like solid lubricants, waxes or other known conventional thickeners.

[0008] Lubricant grease compositions based on calcium sulfonate-based thickener and biodegradable base oil such as polyol ester or polyalkylene glycol has been disclosed in US patent No. 2004/0235679 A1. This patent disclosed the semi-fluid in NLGI 00 / 000 compositions having specific gravity > 1.0, for marine applications where lubricant submerged when

dispensed on water avoiding surface sheen and biodegrade when submerged in water claiming ecology friendly lubricant. In the preferred embodiment where the calcium sulfonate is used 10-20 % alongwith other additives resulted into semi-fluid type of lubricant in NLGI 00 consistency range with penetration range of 400-430 as per ASTM D 217 and without disclosing any other, but desired, performance characteristics of the resultant grease. In general, for any bearing application, greases of atleast NLGI 1 consistency are required and therefore this invention disclosure does not serve the purpose of high-performance bearing grease.

[0009] US Patent No. 2011 / 0111995 A1, disclosed grease composition containing calcium sulfonate complex with wax, preferably carnauba wax, (1:2) ratio in biodegradable oil and water used in preventing welding spatters from adhering to an object to be welded such as steel product in addition to its anti-rust, anti-corrosion properties in an emulsion form and not necessarily used as lubricant. In embodiment 1, calcium sulfonate grease is prepared using either mineral or synthetic oil in combination of biodegradable oil where biodegradable oil is questionable Polyalphaolefin oil or XHVI oil. In this patent disclosures, though the use of biodegradable oil is mentioned, however the base oils used are in combination with either mineral or synthetic oil and the resultant properties of the grease are not clearly meant to be for lubrication purpose.

[0010] US 5,338,467 discloses a process of forming a non-Newtonian oil composition in the form of a grease comprising an overbased calcium sulfonate and solid particles of colloiddally dispersed calcium carbonate in the form of calcite which comprises heating overbased calcium sulfonate, amorphous calcium carbonate and a converting agent comprising a fatty acid of twelve to twenty-four carbon atoms in an oleaginous medium. The oil utilized is a mineral oil.

[0011] US 7,294,608 B2 disclosed a calcium sulfonate grease composition using calcium sulfonate complex thickener, solid lubricants using synthetic, mineral and/or vegetable oils with other performance enhancing additives as thread compounds. The vegetable oils used in the composition could be either seed based or of animal origin. However, the preparation of calcium sulfonate complex grease in vegetable or renewable oils has not been described in the detailed embodiment.

[0012] US 8,618,028 B2 disclosed grease thickener composition based on mixed calcium sulfonate and lithium-based soap in mineral, synthetic, vegetable oil and combination thereof as fire resistant fluid, where commercially available calcium sulfonate as one component was used. This patent does not disclose any composition details of making calcium sulfonate grease in vegetable/biodegradable/ renewable oil. Sagar etal (International Journal of Recent Technologies in Mechanical and Electrical Engineering, Vol 4, Issue 4, p 1- 5, 2017) reported the synthesis of calcium sulfonate grease in soybean oil. In the disclosed process, soybean oil was mixed with 300 TBN sulfonate, calcium carbonate and oleic acid in beaker and the mixture was constantly stirred until the cream color solution was obtained. The solution was put in preheated oven @ 100 °C for 15 minutes. Acetic acid was added which was reported to act as catalyst. The mixture was then subjected to 180 °C heat for 20 minutes followed by addition of calcium hydroxide and boric acid. This mixture was further heated to 200 °C for 60 minutes. The final product was reported to have greasy mixture. An alternative manufacturing process was also suggested using alternate heating technologies such as microwave. The final product was reported to have penetration of 284 i.e., NLGI 2 consistency but low drop point of 105 °C which in today's severe industrial environment do not find much applications as the drop points of final product is much lower than conventionally known for calcium sulfonate greases.

[0013] CN106590814A discloses a thickening agent for complex calcium sulfonate grease, characterized in that it comprises: 21 ~ 50 parts by weight of overbased sulfonates; 0.3 to 5 parts by weight of calcium-containing inorganic substance; Sulfur-containing organic matter 0.01 to 8 parts by weight; The sulfur-containing organic compounds include sulfurized alkylphenols and / or sulfurized alkylphenol phenates. US5207935A discloses a wheel bearing grease, comprising: from about 20% to about 95% of a base oil; a thickener; a succinate-containing texture improving additive for imparting a substantially smooth texture to said grease; a carbonate selected from the group consisting of a carbonate of a Group 1a alkali metal and a carbonate of a Group 2a alkaline earth metal; and a phosphate selected from the group consisting of a phosphate of a Group 1a alkali metal and a phosphate of a Group 2a alkaline earth metal.

[0014] US 2019/203149 A1 discloses a grease composition with improved water resistance and mechanical stability at high-temperatures, the composition comprising: at least one base oil; a water insoluble thickener; and a low molecular weight thixotropic polyamide composition having molecular weight distribution characteristics meeting the requirement described by the relationship: $(A+D)(B+C) \geq 0.05$ where A=the % mass of the polyamide composition with a molecular weight greater than 1700 AMU; B=the % mass of the polyamide composition with a molecular weight between 1100 AMU and 1300 AMU; C=the % mass of the polyamide composition with a molecular weight between 700 AMU and 1000 AMU; and D=the % mass of the polyamide composition with a molecular weight of 600 AMU or lower (determined by GPC).

[0015] US2019338211A1 discloses a hydrocarbon mixture in which: a. the percentage of molecules with even carbon number is $\geq 80\%$ according to FIMS; b. the BP/BI ≥ -0.6037 (Internal alkyl branching per molecule)+2.0 c. on average there are 0.3 to 1.5 5+ methyl per molecule.

[0016] Calcium sulfonate greases compositions and their process of preparations thereof reported in the prior art are either prepared with blend of base oils where one of the oils could either be mineral or synthetic base oil like PAO. Or calcium sulfonate grease is prepared with assistance of well-known thickeners like solids lubricants, calcium carbonate, wax or soap thickeners. The main challenge of making calcium sulfonate greases in these renewable oils is that during the

gelation or conversion of newtonian overbased calcium sulfonate to non-newtonian overbased calcium sulfonate, the reactants tend to react by itself to the base oil and needed conversion of amorphous calcium carbonate to crystalline calcite, as essential step for thickening does not take place adequately. And therefore, the compositions disclosed in prior art, either the combination of base oil is used or combination of other known thickeners has been used to meet desired consistency.

SUMMARY OF THE INVENTION

[0017] The invention is defined in the appended claims. An

embodiment of the invention is a non-Newtonian high performance Overbased Calcium Sulfonate Complex (OBCSC) grease composition prepared in renewable base oil as described herein.

[0018] Another embodiment is grease compositions based on overbased calcium sulfonate complex, lithium, aluminum complex, clay base or polyurea in combination with a renewable base oil (RBO) and no performance additives. These RBO grease compositions exhibit superior performance in lifespan, anti-wear, anti-friction, oxidation resistance, low noise properties, high temperature and pressure characteristics when compared to identical greases prepared in mineral (600 N & 600 R) and PAO 8.

[0019] A further embodiment is zinc free anti-wear grease compositions in renewable base oils.

BRIEF DESCRIPTION OF THE DRAWINGS

[0020]

FIG. 1 is a plot of four ball wear with coefficient of friction with lithium grease in PAO 8, method ASTM D2266.

FIG. 2 is a plot of four ball wear with coefficient of friction with SynNova renewable base oil as described herein, method ASTM D2266.

DETAILED DESCRIPTION OF THE INVENTION

[0021] Overbased calcium sulfonate greases have been an established grease in the state of the art. US 9,273,265 teaches that a known process for making such greases is a two-step process involving the steps of "promotion" and "conversion." Typically the first step ("promotion") is to react a stoichiometric excess amount of calcium oxide (CO) or calcium hydroxide (Ca(OH)₂) as the base source with an alkyl benzene sulfonic acid, carbon dioxide (CO₂), and with other components to produce an oil-soluble overbased calcium sulfonate with amorphous calcium carbonate dispersed therein. These overbased oil-soluble calcium sulfonates are typically clear and bright and have Newtonian rheology. In some cases, they may be slightly turbid, but such variations do not prevent their use in preparing overbased calcium sulfonate greases.

[0022] Typically, the second step ("conversion") is to add a converting agent or agents, such as propylene glycol, isopropyl alcohol, water, formic acid or acetic acid, to the product of the promotion step, along with a suitable base oil such as mineral oil), to convert the amorphous calcium carbonate to a very finely divided dispersion of crystalline calcium carbonate. Because an excess of calcium hydroxide or calcium oxide is used to achieve overbasing, a small amount of residual calcium oxide or calcium hydroxide may also be present and will be dispersed. The crystalline form of the calcium carbonate is preferably calcite. This extremely finely divided calcium carbonate, also known as a colloidal dispersion, interacts with the calcium sulfonate to form a grease-like consistency. Such overbased calcium sulfonate greases produced through the two-step process have come to be known as "simple calcium sulfonate greases" and are disclosed, for example, in U.S. Pat. Nos. 3,242,079; 3,372,115; 3,376,222, 3,377,283; and 3,492,231.

[0023] Additionally, US 4,560, 489 teaches a process and composition for making overbased calcium sulfonate complex (OBCSC) based grease by complexing simple overbased calcium sulfonates by *in situ* reaction of one or more than one inorganic and or organic acids.

[0024] There has been ever increasing demand for high performance lubricating greases based on renewable base oils specially in environment sensitive applications. These high-performance requirements, in general, are extreme pressure & anti-wear, high temperatures, rust & corrosion, water resistance, thermal and oxidative stability etc. Conventionally these environment friendly / biodegradable lubricating greases are prepared either in vegetable oils or their synthetically derived esters. These base oils are essentially esters / glycerides of long chain fatty acids derivatives as compared to hydrocarbons based non-renewable mineral and synthetic base oils. The basic difference in these non-renewable minerals and their synthetically derived base fluids like Polyalphaolefin (PAO's) is that these oils are essentially non-polar whereas vegetable and synthetic esters derived from plant / animal sources are polar in nature which makes lot of difference while formulating overbased calcium sulfonate in these fluids.

[0025] Herein is taught a two-step process of forming a grease composition based on promotion and conversion as

described above wherein a renewable base oil is used in the conversion step of preparing an overbased calcium sulfonate complex grease composition.

[0026] Another embodiment is grease compositions containing the renewable base oil described herein as a component to thickeners utilizing overbased calcium sulfonate complex, lithium, aluminum complex, clay base or polyurea in the grease composition without the use of performance additives that exhibit superior performance in lifespan, anti-wear, anti-friction, oxidation resistance, low noise properties, high temperature and pressure characteristics when compared to identical greases prepared in mineral (600 N & 600 R) and PAO 8.

[0027] A further embodiment is a zinc free anti-wear grease composition in renewable base oils. OBSC grease compositions may need at least 1 % Zinc based anti-wear additive (ZDDP) particularly when OBCSC and lithium greases are prepared in mineral / synthetic PAO oil.

[0028] The different grease compositions have been prepared based on overbased calcium sulfonate complex, lithium, aluminum complex or clay base greases in renewable oil For the purpose of comparison, the same thickener-based grease compositions are also prepared in mineral and synthetic oils.

[0029] For the purposes of this disclosure, the terms "overbased oil-soluble calcium sulfonate" and "oil-soluble overbased calcium sulfonate" and "overbased calcium sulfonate" refer to any overbased calcium sulfonate suitable for making calcium sulfonate greases. The overbased calcium sulfonate content of said greases, as produced by the processes described herein and shown by the illustrative particular examples which are set out below, may be present up to 65% depending on the preferred hardness of the grease. In general grease application rate from NLGI 000, fluid type grease used in gears, to NLGI 3- harder. NLGI 000-NLGI 0 grades are used in gears and whereas NLGI 1-3 for bearing applications. NLGI 000 grease can go as low as 10 % and for NLGI 3 grease it may go up to about 65%. In particular, up to ~ 40 % is for NLGI # 2 grease - medium harness.

[0030] Additionally, as disclosed herein the term "additive" refers to which do not take part in the grease reaction process and remain suspended in the grease matrix and are added to meet certain performance properties.

Renewable Base Oil Component of Grease Composition

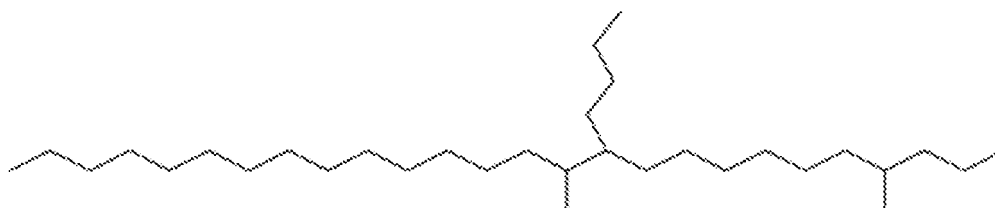
[0031] Renewable base oils used in this invention is understood to be derived from biological resources to be used in making lubricating greases and include the hydrocarbon mixtures described below. Such base oil may be made, but not limited to, from biological organisms designed to manufacture specific oil, but do not include petroleum distilled processed oils such as non-limiting example mineral oils. A suitable method to assess the renewable content is through ASTM D 6866-12, "standard test method for determining biobased content of solid, liquid and gaseous samples using radiocarbon analysis".

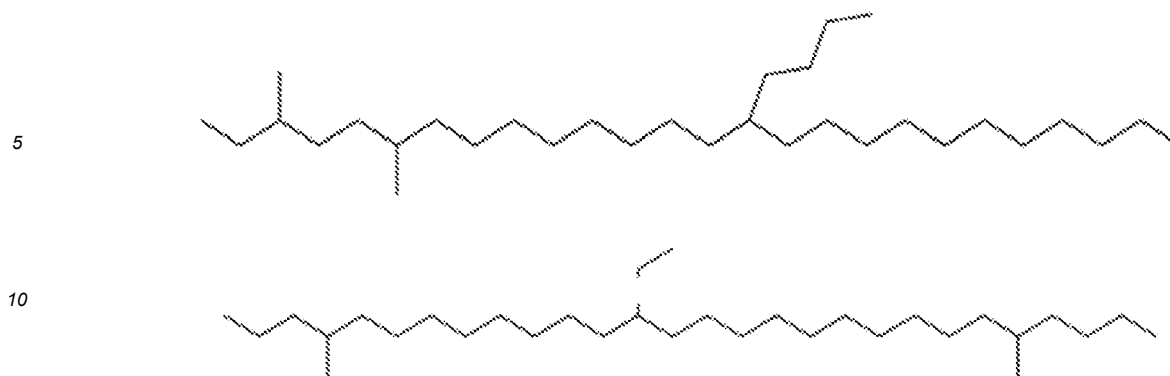
[0032] The renewable base oil described herein and shown by the illustrative particular examples which are set out below, may be present up to about 85%. generally, fall within the weight range of about 85%. In particular, referring to NLGI greases above, NLGI 000 grease with 10 % thickener, the base oil could be about 85 % and for NLGI 3 grease where thickener claim is 65 % the base oil could be as low as 30 %.

[0033] Another embodiment is the use of renewable base oils with the following hydrocarbon structure and properties, additionally referred to herein as "SynNova 9":

The unique branching structure of the hydrocarbon mixtures disclosed herein are characterized by NMR parameters, such as BP, BI, internal alkyl branching, and 5+ methyls. BP/BI of the hydrocarbon mixtures are in the range of ≥ -0.6037 (Internal alkyl branching per molecule) + 2.0. The 5+ methyls of the hydrocarbon mixtures average from 0.3 to 1.5 per molecule.

[0034] The hydrocarbon mixture can be classified into two carbon ranges based on the carbon number distribution, C28 to C40 carbons, and greater than or equal to C42. Generally, about or greater than 95% of the molecules present in each hydrocarbon mixture have carbon numbers within the specified range. Representative molecular structures for the C28 to C40 range can be proposed based on the NMR and FIMS analysis. Without wishing to be bound to any one particular theory, it is believed that the structures made by oligomerization and hydroisomerization of olefins has methyl, ethyl, butyl branches distributed throughout the structure and the branch index and branch proximity contribute to the surprisingly good low temperature properties of the product. Exemplary structures in the present hydrocarbon mixture are as follows:





[0035] The hydrocarbon mixtures exhibit:

- a KV100 in the range of 3.0 - 10.0 cSt;
- a pour point in the range of -20 to -55°C;
- a Noack and CCS at -35°C relationship such that Noack is between $2750 (\text{CCS at } -35^\circ\text{C})^{(-0.8)} \pm 2$;

More preferably the hydrocarbon mixtures have a Noack and CCS at -35 C relationship such that the Noack is between $\text{Noack} = 2750 (\text{CCS at } -35^\circ\text{C})^{(-0.8)} + 0.5$ and $\text{Noack} = 2750 (\text{CCS at } -35^\circ\text{C})^{(-0.8)} - 2$.

[0036] A hydrocarbon mixture in accordance with the present invention with carbon numbers in the range of C28 to C40, and in another embodiment carbon numbers in the range of from C28 to C36, or in another embodiment molecules with a carbon number of C32, will generally exhibit the following characteristics in addition to the characteristics of BP/BI, Internal alkyl branches per molecule, 5+ methyl branches per molecule, and Noack/CCS relationship described above:

- a KV100 in the range of 3.0 - 6.0 cSt;
- a VI in the range of $11 \ln(\text{BP/BI}) + 135$ to $11 \ln(\text{BP/BI}) + 145$; and
- a pour point in the range of $33 \ln(\text{BP/BI}) - 45$ to $33 \ln(\text{BP/BI}) - 35$.

[0037] In one embodiment, the KV100 for the C28 - C40 hydrocarbon mixture ranges from 3.2 to 5.5 cSt; in another embodiment the KV100 ranges from 4.0 to 5.2 cSt; and from 4.1 to 4.5 cSt in another embodiment.

[0038] The VI for the C28-C40 hydrocarbon mixture ranges from 125 to 155 in one embodiment and from 135 to 145 in another embodiment.

[0039] The Pour Point of the hydrocarbon mixture, in one embodiment ranges from 25 to -55°C and from 35 to -45°C in another embodiment.

[0040] The boiling point range of the C28-C40 hydrocarbon mixture in one embodiment is no greater than 125°C (TBP at 95% - TBP at 5%) as measured by ASTM D2887; no greater than 100°C in another embodiment; no greater than 75°C in one embodiment; no greater than 50°C in another embodiment; and no greater than 30°C in one embodiment. In the preferred embodiments, those with a boiling point range no greater than 50 °C, and even more preferably no greater than 30°C, give a surprisingly low Noack Volatility (ASTM D5800) for a given KV100.

[0041] The C28 - C40 hydrocarbon mixture in one embodiment has a Branching Proximity (BP) in the range of 14-30 with a Branching Index (BI) in the range of 15 - 25; and in another embodiment a BP in the range of 15 - 28 and a BI in the range of 16 - 24.

[0042] The Noack volatility (ASTM D5800) of the C28 - C40 hydrocarbon mixture is less than 16 wt% in one embodiment; less than 12 wt% in one embodiment; less than 10 wt% in one embodiment; less than 8 wt% in one embodiment and less than 7 wt% in one embodiment. The C28 - C40 hydrocarbon mixture in one embodiment also has a CCS viscosity at -35°C of less than 2700 cP; of less than 2000 cP in another embodiment; of less than 1700 cP in one embodiment; and less than 1500 cP in one embodiment.

[0043] The hydrocarbon mixture with the carbon number range of C42 and greater will generally exhibit the following characteristics, in addition to the characteristics of BP/BI, internal alkyl branches per molecule, 5+ methyl branches per molecule, and Noack and CCS at -35°C relationship described above:

- a KV100 in the range of 6.0 - 10.0 cSt;
- a VI in the range of $11 \ln(\text{BP/BI}) + 145$ to $11 \ln(\text{BP/BI}) + 160$; and
- a Pour Point in the range of $33 \ln(\text{BP/BI}) - 40$ to $33 \ln(\text{BP/BI}) - 25$.

[0044] The hydrocarbon mixture comprising C42 carbons or greater, in one embodiment has a KV100 in the range of 8.0

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to 10.0 cSt, and in another embodiment from 8.5 to 9.5 cSt.

[0045] The VI of the hydrocarbon mixture having ≥ 42 carbons is 140 - 170 in one embodiment; and, from 150 - 160 in another embodiment.

[0046] The pour point in one embodiment ranges from -15 to -50°C; and, from -20 to -40°C in another embodiment.

[0047] In one embodiment, the hydrocarbon mixture comprising ≥ 42 carbons has a BP in the range of 18 - 28 with a BI in the range of 17 - 23. In another embodiment, the hydrocarbon mixture has a BP in the range of 18 - 28 and a BI in the range of 17 - 23.

[0048] In general, both hydrocarbon mixtures disclosed above exhibit the following characteristics:

- at least 80% of the molecules have an even carbon number according to FIMS;
- a KV100 in the range of 3.0 - 10.0 cSt;
- a pour point in the range of -20 to -55°C;
- a Noack and CCS @ -35°C relationship such that Noack is between $2750 (\text{CCS @ -35°C})^{(-0.8)} \pm 2$;
- a BP/BI in the range of ≥ -0.6037 (Internal alkyl branching) + 2.0 per molecule; and,
- on average from 0.3 to 1.5 5+ methyl branches per molecule.

[0049] High performance grease: a grease capable of working a high and low temperatures, under heavy loads, mechanical and shear stable, rust and corrosion protection and stable under influence of water and humid atmosphere, suitable high and slow speeds. For the sake of better understanding, high performance characteristics of a grease can be described as in following table

Table - 1*

	ASTM test method	Multi-purpose	High performance
<u>Stability / life</u>			
Roll stability, 2 hrs; %	D 1831	≥ 10	< 10
High temp life @ 160; hrs	D 3527	< 80	> 80
<u>Operating temperature & Oxdn stability</u>			
Oxidation resistance	D 942	≥ 10	< 10
Operating temperature range, °F	-	0 to 250 F	0 to 350
<u>Load carrying / wear protection</u>			
Weld load, kg	D 2596	< 250	> 250
Wear scar dia, mm	D 2266	≥ 0.6	≤ 0.6
Fretting wear, mg	D 4170	-	< 10
Coefficient of friction	D 5707	≥ 0.1	< 0.1
<u>Water resistance</u>			
Water washout, 79 C; %	D 1264	≥ 5	< 5
Rust & corrosion protection	D 1743	Pass	pass
<i>*for reference only</i>			

[0050] The consistency of the the grease compositions have been tested as ASTM D 217, "standard test methods for cone penetration of lubricating grease". The cone penetration provides the measure of a grease consistency / hardness under specified test conditions. The stability of grease compositions has been evaluated with testing the roll stability of the greases as tested by ASTM D 1831-19, "standard test method for roll stability of lubricating greases". This test method provides the changes in the consistency, as measured by cone penetration, of lubricating greases under rolling shear when worked in the roll stability test apparatus. Lower the change in difference in penetration before and after the test,

better is the stability of the grease under rolling shear. High temperature life of the grease compositions has been tested by ASTM D 3527-18, "standard test method for life performance of automotive wheel bearing grease". The test grease is distributed in the bearings of an automobile front wheel hub-spindle-bearings assembly. The bearings are thrust-loaded to approximately 111 N, the hub is rotated at 1000 r/min and the spindle temperature maintained at 160 °C for 20 h, 4 h off operating cycle. The test is terminated when grease deterioration causes the drive motor torque to exceed a calculated motor cut off value. Grease life is expressed as the accumulated on-cycle hours. Higher the test hours, better the high temperature life of the grease. As per NLGI GC-LB specification as specified in ASTM D 4950, "standard classification and specification for automotive service Greases" the high temperature life requirement to meet this specification in minimum 80 hours. The high temperature life of ball bearings has further been evaluated as per ASTM D 3336-18, "standard test method for life of lubricating greases in ball bearings at elevated temperatures". A grease lubricated SAE No. 204 size ball bearing is rotated at 10 000 r/min under thrust load of 22 N 6 2 N (5 lbf 6 0.55 lbf) applied to the outer race of the bearing by means of a helical spring and at a 177 °C temperature. Tests are continued until failure or completion of a specified number of hours of running time. Higher the hours to bearing failure, higher is the life of grease under test conditions.

[0051] Thermal and oxidation stability of the grease compositions described in this invention are tested as per ASTM D 942-15, "standard test method for oxidation stability of lubricating greases by the oxygen pressure vessel method". The sample of grease is oxidized in a pressure vessel heated to 99 °C (210 °F) and filled with oxygen at 110 psi (758 kPa). Pressure is observed and recorded at stated intervals. The degree of oxidation after 100 hours is determined by the corresponding decrease in oxygen pressure and recorded as psi drop. The oxidation stability of the grease samples is also measured by pressure differential Scanning Calorimetry (PDSC) as per ASTM D 5483, "oxidation induction time of lubricating greases by pressure differential scanning calorimetry". A small quantity of grease is weighed into a sample pan and placed in a test cell. The cell is heated to a specified temperature (155 °C, 180 °C and 210 °C) and then pressurized with oxygen. The cell is held at a regulated temperature and pressure until an exothermic reaction occurs. The extrapolated onset time is measured and reported as the oxidation induction time for the grease under the specified test temperature. Oxidation induction time, as determined under the conditions by this test method, can be used as an indication of oxidation stability. Higher the oxidation induction time, better is the oxidation stability of the grease under test conditions.

[0052] Anti-friction, load carrying, and wear protection properties of the grease compositions have been evaluated as per ASTM D 2596-15, "standard test method for measurement of extreme-pressure properties of lubricating grease (four-ball method)" and ASTM D 2266-01 (reapproved 2015), "standard test method for wear preventive characteristics of lubricating grease (four-ball method)". Three ½ inch (12.7 mm) diameter steel balls are clamped together and covered with the lubricant to be evaluated. A fourth ½ inch diameter steel ball, referred to as the top ball, is pressed with a force of 40 kgf (392 N) into the cavity formed by the three clamped balls for three-point contact. The temperature of the lubricating grease specimen is regulated at 75 °C (167 °F) and then the top ball is rotated at 1200 rpm for 60 min. Lubricants are compared by using the average size of the scar diameters worn on the three lower clamped balls. This method was further extended to evaluate coefficient of friction under these specified conditions.

[0053] The overbased calcium sulfonate has a total base number (TBN) of 300 to 450 mgKOH/g in mineral, white or synthetic hydrocarbon diluent.

[0054] To facilitate the conversion process, promoters for conversion of amorphous calcium carbonate to calcite such as propylene glycol, C1-3 alcohols, C1-5 monocarboxylic acids, water, methyl Cellosolve as such or a combination thereof may be used.

[0055] To facilitate the process of complexing one or more of the inorganic acids containing either boron or phosphorous as exemplified as boric and phosphoric acids, organic acids containing aromatic acids exemplified as salicylic acid, mono carboxylic acid exemplified as acetic acid, dicarboxylic acids exemplified as azelaic acid and long chain fatty acids containing atleast C 12 carbon atoms as exemplified as 12-hydroxy stearic acid may be used.

[0056] In order to fully describe the the nature of present invention, specific examples will hereinafter be described. It should be understood, however, that this is done solely by way of example and is intended to delineate not limit the ambit of the appended claims.

EXAMPLES

Example 1.

[0057] In this example novel composition of overbased calcium sulfonate complex (OBCSC) grease using renewable base oil for the first time has been disclosed as in table 2.

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Table-2

Component	Weight %
SynNova 9	55.05
Overbased calcium sulfonate (Lubrizol GR9251)	38.05
Water	11.00
Calcium hydroxide	2.21
Boric acid	2.04
12-hydroxy stearic acid	2.65

120 gm of base oil was charged in a kitchen aid mixer and 430 gm of newtonian overbased calcium sulfonate (LZ GR9251) was added to this with continuous stirring. To this mixture 60 gm of water was added and mixed continuously for 45-60 minutes. The heat is continuously and slowly providing to the mixer and temperature was raised to 180 -190 °F. To this mixture 120 gm of oil was added. In a separate pot, slurry of 20.40 gm of boric acid, 22.10 gm of calcium hydroxide, and 50 gm of water was made and then added to this mixture. 120 gm of oil was added to this mixture. The mass was heated to 200-220 °F and then 26.50 gm of 12-hydroxy stearic acid was added. The temperature was gradually increased to 300-320 °F. Subsequently, heat was turned off and mass was gradually cooled down, balance base oil added and mill through 3 roll mills. This disclosed composition does not contain any performance additives. The resultant grease exhibited following properties as shown in table-3.

Table-3

#	Properties	Test Method	Test Results
1.	Thickener %		44.95
2.	Penetration	ASTM D 217	309
3.	Roll Stability, 2 hrs, % change	ASTM D 1831	1.29
4.	Drop point, °F	ASTM D 2265	+ 600
5.	Water washout, % wt.	ASTM D 1264	1.75
6.	Four ball weld loads, kgs	ASTM D 2596	295
7.	Four ball wear scar diameters, mm	ASTM D 2266	0.324
8.	Coefficient of friction	ASTM D 2266	0.084
9.	Fretting wear protection, gm	ASTM D 4170	0.6
10.	Life performance of automotive wheel bearing grease, hrs	ASTM D 3527	85.7
11.	Life of lubricating greases in ball bearings at elevated temperatures, hrs	ASTM D 3336	64.4
12.	Oxidation stability of lubricating greases by the oxygen pressure vessel method, 100 hrs, psi drop	ASTM D 942	1.1
13.	Oxidation induction time of lubricating greases by PDSC, 180 °C	ASTM D 5483	19.9
14.	Bearing noise	ANDEROMETER®	4.9

[0058] As per table-3 , the test data indicate that the grease disclosed in this embodiment without any performance additive exhibit excellent stability as indicated by only 1.29 % change roll stability , very high prop point of + 600 °F, 295 kg weld load thus high load carrying capability , excellent anti-wear properties as indicated by low wear scar diameter of only 0.324 mm and only 0.6 mg fretting wear , excellent anti-friction properties as indicated by low coefficient of friction of 0.084, excellent high temperature life of 85.7 hrs at 160 °C (ASTM D 3527) and 64.4 hrs at 177 °C (ASTM D 3336), excellent oxidation stability as indicated by only 1.1 psi drop after 100 hrs (ASTM D 942) and 19.9 minute oxidation induction time at 180 °C (ASTM D 5483) and thus high potentials to be high performance grease for wide range of applications.

[0059] This grease can also alternately be prepared using other newtonian overbased sulfonate sulfonates known to one of skill in the art supplied by Lubrizol (LZ 75 NS, LZ 75 GR, LZ 75 WR, LZ 75 P), Lockard, Oronite, Chemtura, Daubert chemicals with similar or other alternate process.

Example 2:

[0060] In this example the grease compositions were prepared using practically identical composition and process as described in example 1 but in place of SynNova 9, the base oil used to prepare grease was synthetic Polyalphaolefin in 8 cSt viscosity @ 100 °C (PAO-8) oil. The resultant grease properties have been compared with grease prepared with SynNova 9 as per example 1 and test results are tabulated in table-4

Table-4

#	Properties	Test Method	OBCSC grease with SynNova 9	OBCSC grease with PAO-8
1.	Thickener %		44.95	47.62
2.	Roll Stability, 2 hrs, % change	ASTM D 1831	1.29	1.64
3.	Bearing Noise	Anderometer	4.9	5.6
4.	Four ball weld loads, kgs	ASTM D 2596	295	275
5.	Four ball wear scar diameters, mm	ASTM D 2266	0.324	0.422
6.	Fretting wear protection, gm	ASTM D 4170	0.6	4.4
7.	Life performance of automotive wheel bearing grease	ASTM D 3527	85.7	40.4
8.	Life of lubricating greases in ball bearings at elevated temperatures	ASTM D 3336	64.4	53
9.	Oxidation stability of lubricating greases by the oxygen pressure vessel method, 100 hrs, psi drop	ASTM D 942	1.1	1.6
10.	Oxidation induction time of lubricating greases by PDSC, 180 °C	ASTM D 5483	19.9	16.6

[0061] Table-4 clearly indicate that OBCSC grease prepared in renewable SynNova 9 exhibited less thickener content compared to same grease prepared with same composition and process in PAO-8. Less thickener content is indicative of better dispersibility/ solubility of overbased calcium sulfonate thickener in base oil. Solubility / dispersion of thickener in base oil plays significantly important role in lubricating greases in guiding their performance characteristics like consistency, mechanical/ shear stability and bearing noise as can be seen from better roll stability of 1.29 % in case of grease with SynNova 9 oil based compared to 1.64 % in case of grease prepared with PAO 8 and tested as per ASTM D 1831, and comparatively low bearing noise of 4.9 with SynNova 9 based grease and 5.6 with grease based on PAO 8 as tested by ANDEROMETER®.

[0062] As indicated in table-4, OBCSC grease prepared in SynNova 9 exhibited higher load carrying capabilities as indicated by higher weld load of 295 kg, compared to 275 kg of OBCSC grease prepared with PAO-8 base oil.

[0063] Anti-wear properties of OBCSC grease prepared with SynNova 9 exhibited much superior wear scar of 0.324 mm compared to corresponding OBCSC grease in PAO-8 with 0.422 mm tested as per ASTM D 2266. The similar anti-wear performance properties of OBCSC grease prepared in example 1 with SynNova 9 only exhibited 0.6 mg fretting wear compared to corresponding 4.4 mg with OBCSC grease prepared in PAO-8; tested as per ASTM D 4170.

[0064] High temperature life of OBCSC grease prepared in example 1 with SynNova 9 exhibited 85.7 hrs compared to only 40.4 hrs with identical OBCSC grease with PAO-8 tested as per ASTM D 3527 @ 160 °C. This observation is further supported by the fact that the life of the OBCSC grease with SynNova 9 base oil, tested in ball bearing at 177 °C (ASTM D 3336) came out to be 64.4 hrs compared to identical OBCSC grease prepared in this example with PAO-8 with less life of 53 hrs.

[0065] The oxidation resistance properties of the two greases prepared in example 1 and example 2 were compared as per ASTM D 942 and ASTM D 5483. The OBCSC grease prepared in example 1 with SynNova 9 base exhibited pressure drop after 100 hrs @ 100 °C of only 1.1 psi compared to OBCSC grease in PAO-8 with pressure drop of 1.6 psi, thus lower pressure prop of grease with SynNova 9 oil indicate superior oxidation resistance. This is observation is further supported by testing the grease by PDSC at 180 °C (ASTM D 5483) where OBCSC grease with SynNova 9 exhibited higher 19.9-minute induction time compared to 16.6 minutes with OBCSC grease in PAO-8.

Example 3

[0066] In this example, the grease compositions were prepared identical as described in example 1 but in place of SynNova 9, the base oil used to prepare was mineral oil; 600 N. The properties of resultant grease are compared in table-5 with grease prepared with SynNova 9 as described in example 1

Table-5

#	Properties	Test Method	OBCSC grease with SynNova 9	OBCSC Grease with 600 N
1.	Thickener %		44.95	46.18
2.	Bearing Noise	Anderometer	4.9	7.7
3.	water washout characteristics of lubricating grease, 79 °C, % wt.	ASTM D 1264	1.75	2.00
4.	Four ball wear scar diameters, mm	ASTM D 2266	0.324	0.413
5.	Fretting wear protection, gm	ASTM D 4170	0.6	4.40
6.	Life performance of automotive wheel bearing grease	ASTM D 3527	85.7	40.0
7.	Oxidation stability of lubricating greases by the oxygen pressure vessel method, 100 hrs, psi drop	ASTM D 942	1.1	3.2

[0067] Table-5 clearly indicate that OBCSC grease prepared in renewable SynNova 9 exhibited less thickener content of 44.95 % compared to same grease prepared with same composition and process in 600 N base with 46.8 %. The bearing noise as tested by Anderometer came out be 4.9, better with grease made in SynNova 9 oil compared to 7.7 with grease prepared in 600 N oil. The water washout characteristics of grease tested as per ASTM D 1264 and prepared with SynNova 9 base oil exhibited water washout of 1.75 % compared to 2.0 % in case of identical grease prepared in 600 N base oil. Lower the water washout, better is the water resistance characteristics of the grease.

[0068] Anti-wear properties of OBCSC grease prepared with SynNova 9 exhibited much superior wear scar diameter of 0.324 mm compared to corresponding OBCSC grease in 600 N with 0.413 mm tested as per ASTM D 2266. The similar anti-wear performance properties of OBCSC grease prepared in example 1 with SynNova 9 only gave 0.6 mg fretting wear compared to corresponding 4.4 mg with OBCSC grease prepared in 600 N; tested as per ASTM D 4170

[0069] High temperature life of OBCSC grease prepared in example 1 with SynNova 9 exhibited 85.7 hrs compared to only 40.0 hrs with identical OBCSC grease with 600 N tested as per ASTM D 3527 @ 160 °C.

[0070] The oxidation resistance properties of the two greases prepared in example 1 and example 3 were compared as per ASTM D 942. The OBCSC grease prepared in example 1 with SynNova 9 base exhibited pressure drop after 100 hrs @ 100 °C of only 1.1 psi compared to identically prepared OBCSC grease as per example 3 with 600 N with pressure drop of 3.2 thus lower pressure drop of grease with SynNova 9 oil indicate superior oxidation resistance.

Example 4

[0071] In this embodiment, lithium 12 hydroxy stearate grease is prepared by conventional open kettle process by taking 158.6 gm of SynNova 9 in single rotating, medium speed kitchen mixer. To this mixer 126.8 gm of 12 hydroxy stearic acid was added and heated till melting. To this mixture, 19.0 gm of lithium hydroxide monohydrate slurry in water was added and 200 gm of SynNova 9 oil was then added. The temperature was gradually increased to 400 °F with continuous stirring. At this temperature, heat was turned off and 495.6 gm of SynNova 9 was further added. The mass was cooled down to < 180 °F and mill through 3 roll mill and tested for the tests as indicated in table-6. For comparison purpose other lithium greases were also prepared using same raw materials and identical process using group II mineral oil; Chevron 600 R and synthetic Polyalphaolefin 8 cSt base oil. The comparative test data are tabulated in following table 6

Table-6

#	Properties	Test Method	Lithium grease with SynNova 9	Lithium grease with PAO-8	Lithium grease with 600 R
1.	Thickener, %		14.58	16.08	16.13
2.	Roll stability, 2 hrs, % change	ASTM D 1831	1.72	2.69	2.76
3.	water washout characteristics of lubricating grease, 79 °C, % wt.	ASTM D 1264	2.00	2.50	4.5
4.	Bearing Noise	Anderometer	7.7	8.2	8.5
5.	Four ball wear scar diameters, mm	ASTM D 2266	0.552	0.631	0.585
6.	Four ball wear with friction graph, wear scar diameter, mm	ASTM D 2266	0.75	1.01	
	Average Coefficient of Friction		0.088	0.115	
7.	Life of greases in ball bearings at elevated temperatures	ASTM D 3336	73.2	60.0	
8.	Oxidation stability of grease by the oxygen pressure vessel method, 100 hrs, psi drop	ASTM D 942	72.5	95.4	99.3
9.	oxidation induction time of Greases by PDSC, 155 °C, minutes	ASTM D 5483	< 10	21.4	

[0072] Table-6 indicate that lithium grease prepared in renewable SynNova 9 exhibited less thickener content of 14.58 % compared to same grease prepared with same composition and process in PAO 8 base oil with 16.08 % and in 600 R oil with thickener content of 16.13 %. Shell roll stability of grease prepared with renewable SynNova 9 oil is 1.72 % better than grease prepared with PAO 8 and 600 R with 2.69 % and 2.76 % respectively. The average bearing noise of the grease prepared with SynNova 9 is 7.7 better than grease prepared with PAO 8 and 600 R with average noise of 8.2 and 8.5 respectively. The water washout characteristics of lithium grease prepared with SynNova 9 base oil exhibited better water washout of 2.00 % compared to 2.5 %, grease prepared in PAO 8 and 4.5 %, grease prepared in 600 R.

[0073] Anti-wear properties of lithium grease prepared with SynNova 9 exhibited much superior wear scar diameter of 0.552 mm compared to corresponding lithium grease in PAO 8 with 0.631 mm and 0.585 mm for grease prepared in 600 R. This test result was further validated by running the same test on different equipment but with friction curve. The grease with SynNova 9 exhibited wear scar diameter of 0.75 mm compared to 1.01 mm for the grease with PAO 8. The average coefficient of friction of the lithium grease with SynNova 9 base oil came out to be 0.088 with Y-O intercept 0.088 where as in case of lithium grease with PAO 8 was found to be much higher value with average coefficient of friction 0.115 with Y-O intercept of 0.093 as shown in the figure 1 and 2. Figure 1 which corresponds to lithium grease with SynNova 9, clearly stands out with uniform and smooth friction pattern over the test period compared to irregular friction pattern with disrupted film in figure-2, indicating potential metal to metal contact

[0074] High temperature life of lithium grease prepared in SynNova 9 tested in ball bearing at 177 °C as per ASTM D 3336 with test result of 73.2 hrs compared to identical lithium grease prepared in this example with PAO-8 with lower life of only 60 hrs.

[0075] The oxidation resistance properties of the two greases without any additive, prepared in this example were compared as per ASTM D 942 and ASTM D 5483. The lithium grease prepared with SynNova 9 base exhibited pressure drop after 100 hrs @ 100 °C of 72.5 psi compared to identically prepared and tested lithium grease with PAO-8 with pressure drop of 95.4 and 99.3 grease in 600 R oil. This observation is further confirmed by testing the same greases by PDSC at 155 °C (ASTM D 5483) where lithium grease with SynNova 9 exhibited higher 21.4 minutes induction time compared to < 10 minutes in case of lithium grease with PAO-8

Example 5

[0076] In this example, the effect of Zinc dialkyldithiophosphate (ZDDP), a well-known anti-wear additive, on the comparative wear scar dia of all the greases as prepared in example 1,2 and 3 has been presented. All three greases were treated with 1 % LZ 1395; a Lubrizol corporation supplied a commercial zinc dialkyldithiophosphate (ZDDP) with typical

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phosphorous content of 9.50 %, sulphated ash 15.90 % and zinc content of 10.60 %. The test results with and without ZDDP are tabulated in table-7.

Table-7

	Wear scar diameter, mm; ASTM D 2266						
	Type of thickener	Grease with SynNova 9		Grease with PAO 8		Grease with 600 N	
		No Additive	+1 % ZDDP	No Additive	+ 1 % ZDDP	No Additive	+ 1 % ZDDP
1.	OBCSC	0.324	0.446	0.422	0.320	0.413	0.388
2.	Lithium	0.552	0.496	0.631	0.588	0.585	0.525

[0077] OBCSC grease prepared as per example-1 in SynNova base oil exhibited wear scar of 0.324 mm whereas identical OBCSC grease prepared with PAO 8 as per example 2 indicated wear scar diameter of 0.422. When in the same grease was doped with 1 % ZDDP as described above reduced the wear scar diameter to 0.32 mm almost same as wear scar diameter of OBCSC prepared with SynNova 9 that do not contain any additive including ZDDP. Similarly, OBCSC grease prepared as per example 3 in group I mineral oil 600 N oil exhibited wear scar diameter without any anti-wear additive of 0.413 mm. When the same grease was doped with 1 % above described ZDDP, the wear scar diameter is reduced to from 0.413 mm to 0.388 mm. If we compare wear scar diameter of OBCSC made with SynNova 9 without any ZDDP / antiwear additive showed lower wear scar diameter of 0.324 mm compared to identical OBCSC prepared with 600 N oil even with 1 % ZDDP with wear scar diameter of 0.388; higher than OBCSC grease in SynNova 9 oil and without any ZDDP.

Example 6

[0078]

Table-8

PDSC (ASTM D 5483), Oxidation Induction Time, minutes							
Type of thickener	Test temperature	Grease in SynNova 9		Grease in 600 R		Grease in PAO 8	
		Grease + 0.5 % LZ 9510	Grease + 1.0 % LZ 9510	Grease + 0.5 % LZ 9510	Grease + 1.0 % LZ 9510	Grease + 0.5 % LZ 9510	Grease + 1.0 % LZ 9510
OBCSC	210 °C	84.5	> 120	23.4	58.3	60.45	123.2
Lithium Grease	180°C	-	>120	-	-	-	98.4

[0079] In this example, the effect of antioxidant additives on the OBCSC and lithium greases prepared in SynNova 9, PAO 8 and 600 R has been compared. The greases were doped with either 0.5 % or 1.0 % LZ 9510, a substituted diarylamine type oxidation inhibitor with typical nitrogen content of 3.5 % and tested for oxidation induction time at different temperatures by PDSC as per ASTM D 5483. In OBCSC grease in SynNova 9 with 1 % LZ9510 oxidation induction time was > 120 minutes with no isotherm at 210 °C compared to 58.3 minutes in case of OBCSC grease in 600 R with 1 % LZ 9510 and 123.2 minutes in case of grease with PAO 8. In order to get clear distinction, the test was run with all three greases with 0.5 % LZ 9510. OBCSC in SynNova 9 with 0.5 % LZ 9510 gave 84.5 minutes induction time @ 210 °C compared to corresponding 60.45 minutes and 23.4 minutes in case of OBCSC grease with PAO 8 and 600 R respectively. This test was ran at 180 °C on lithium greases with SynNova 9 and PAO 8 and test results were > 120 minutes with lithium grease in SynNova 9 with 1.0 % LZ 9510 and 98.4 minutes with grease in PAO 8 and with 1 % LZ 9510. This example clearly indicate that addition of known anti-oxidants to these greases, greases made in SynNova 9 maintains it superior performance characteristics over the other greases made in PAO 8 or mineral oil based 600 R.

Claims

1. A high-performance non-Newtonian overbased calcium sulfonate complex grease composition comprising over-based calcium sulfonate and a renewable base oil, wherein the renewable base oil comprises a KV100 in the range of

3.0 - 10.0 cSt; a pour point in the range of -20 to -55°C; a Noack and CCS at -35°C relationship such that Noack is between 2750 (CCS at -35°C)^(-0.8) ± 2; and wherein the renewable base oil is the only base oil present in the composition.

2. The composition of claim 1 wherein the renewable base oil comprises the following NMR parameters: (a) BP/BI of the hydrocarbon mixtures are in the range of ≥ -0.6037 with internal alkyl branching per molecule + 2.0 and (b) the 5+ methyls of the hydrocarbon mixtures average from 0.3 to 1.5 per molecule.
3. Use of the composition of claim 2 as an anti-wear composition, wherein no additional performance additive is required.
4. The use of claim 3, wherein the composition does not contain zinc.
5. The process of forming a non-Newtonian oil composition in the form of a grease comprising an overbased calcium sulfonate and a renewable base oil, (1) heating overbased calcium sulfonate, calcium hydroxide and a converting agent, comprising a renewable base oil with a KV100 in the range of 3.0 - 10.0 cSt; a pour point in the range of -20 to -55°C; a Noack and CCS at -35°C relationship such that Noack is between 2750 (CCS at -35°C)^(-0.8) ± 2; (2) reacting said product of step 1 with components comprising a boric acid compound to develop the grease like properties, wherein the renewable base oil is the only base oil used in the process.
6. The process of claim 5 wherein the reaction proceeds in the presence of 12-hydroxy stearic acid.
7. A grease composition comprising a renewable base oil and a thickener wherein the renewable base oil comprises a KV100 in the range of 3.0 - 10.0 cSt; a pour point in the range of -20 to -55°C; a Noack and CCS at -35°C relationship such that Noack is between 2750 (CCS at -35°C)^(-0.8) ± 2; and the thickener is selected from the group consisting of lithium, aluminum complex, clay base and polyurea; and wherein the renewable base oil is the only base oil present in the composition.
8. The composition of claim 7 wherein the renewable base oil comprises the following NMR parameters: (a) BP/BI of the hydrocarbon mixtures are in the range of ≥ -0.6037 with internal alkyl branching per molecule + 2.0 and (b) the 5+ methyls of the hydrocarbon mixtures average from 0.3 to 1.5 per molecule.
9. The composition of claim 8 wherein the composition does not contain an additional performance additive.
10. The composition of claim 8 where in the composition contain additional performance additives.

Patentansprüche

1. Nicht-Newtonsche überalkalisiertes-Calciumsulfonat-Komplex-Hochleistungs-Schmiermittelzusammensetzung, die überalkalisiertes Calciumsulfonat und ein erneuerbares Basisöl umfasst, wobei das erneuerbare Basisöl eine KV100 im Bereich von 3,0 - 10,0 cSt; einen Stockpunkt im Bereich von -20 bis -55 °C; einen Noack und einen CCS in einer solchen Beziehung bei -35 °C umfasst, dass der Noack zwischen 2750 (CCS bei -35 °C)^(-0.8) ± 2 beträgt; und wobei das erneuerbare Basisöl das einzige in der Zusammensetzung vorhandene Basisöl ist.
2. Zusammensetzung nach Anspruch 1, wobei das erneuerbare Basisöl die folgenden NMR-Parameter umfasst: (a) die BP/BI der Kohlenwasserstoffgemische liegen im Bereich von $\geq -0,6037$ mit einer internen Alkylverzweigung pro Molekül von +2,0 und (b) die 5+-Methyle der Kohlenwasserstoffgemische betragen durchschnittlich von 0,3 bis 1,5 pro Molekül.
3. Verwendung der Zusammensetzung nach Anspruch 2 als Verschleißschutz-Zusammensetzung, wobei kein zusätzliches Leistungsadditiv erforderlich ist.
4. Verwendung nach Anspruch 3, wobei die Zusammensetzung kein Zink enthält.
5. Verfahren zur Bildung einer nicht-Newtonschen Ölzusammensetzung in Form eines Schmiermittels, das ein überalkalisiertes Calciumsulfonat und ein erneuerbares Basisöl umfasst, (1) das Erhitzen von überalkalisiertem Calciumsulfonat, Calciumhydroxid und einem Umwandlungsmittel, das ein erneuerbares Basisöl mit einer KV100 im Bereich von 3,0 - 10,0 cSt; einen Stockpunkt im Bereich von -20 bis -55 °C; einen Noack und einen CCS in einer

solchen Beziehung bei -35 °C umfasst, dass der Noack zwischen 2750 (CCS bei -35 °C) $(-0,8) \pm 2$ beträgt; (2) das Umsetzen des Produkts aus Schritt 1 mit Komponenten, die eine Borsäure-Verbindung umfassen, wodurch die schmiermittelartigen Eigenschaften entwickelt werden, wobei das erneuerbare Basisöl das einzige in dem Verfahren verwendete Basisöl ist.

6. Verfahren nach Anspruch 5, wobei die Reaktion in Gegenwart von 12-Hydroxystearinsäure erfolgt.
7. Schmiermittelzusammensetzung, die ein erneuerbares Basisöl und ein Verdickungsmittel umfasst, wobei das erneuerbare Basisöl eine KV100 im Bereich von 3,0 - 10,0 cSt; einen Stockpunkt im Bereich von -20 bis -55 °C; einen Noack und einen CCS in einer solchen Beziehung bei -35 °C umfasst, dass der Noack zwischen 2750 (CCS bei -35 °C) $(-0,8) \pm 2$ beträgt; und das Verdickungsmittel ausgewählt ist aus der Gruppe bestehend aus Lithium, einem Aluminiumkomplex, einer Tonbasis und Polyharnstoff und wobei das erneuerbare Basisöl das einzige in der Zusammensetzung vorhandene Basisöl ist.
8. Zusammensetzung nach Anspruch 7, wobei das erneuerbare Basisöl die folgenden NMR-Parameter umfasst: (a) die BP/BI der Kohlenwasserstoffgemische liegen im Bereich von $\geq -0,6037$ mit einer internen Alkylverzweigung pro Molekül von +2,0 und (b) die 5+-Methyle der Kohlenwasserstoffgemische betragen durchschnittlich von 0,3 bis 1,5 pro Molekül.
9. Zusammensetzung nach Anspruch 8, wobei die Zusammensetzung kein zusätzliches Leistungsadditiv enthält.
10. Zusammensetzung nach Anspruch 8, wobei die Zusammensetzung zusätzliche Leistungsadditive enthält.

Revendications

1. Composition de graisse de complexe de sulfonate de calcium surbasé non newtonien à haute performance comprenant du sulfonate de calcium surbasé et une huile de base renouvelable, dans laquelle l'huile de base renouvelable comprend un KV100 dans la plage de 3,0 à 10,0 cSt ; un point d'écoulement dans la plage de -20 à -55 °C; une relation de Noack et CCS à -35 °C telle que Noack est compris entre 2 750 (CCS à -35 °C) $(-0,8) \pm 2$; et dans laquelle l'huile de base renouvelable est la seule huile de base présente dans la composition.
2. Composition selon la revendication 1 dans laquelle l'huile de base renouvelable comprend les paramètres RMN suivants : (a) BP/BI des mélanges hydrocarbonés sont dans la plage de $\geq -0,6037$ avec ramification d'alkyle interne par molécule + 2,0 et (b) les méthyles 5+ des mélanges d'hydrocarbure sont en moyenne de 0,3 à 1,5 par molécule.
3. Utilisation de la composition selon la revendication 2 en tant que composition anti-usure, dans laquelle aucun additif de performance supplémentaire n'est requis.
4. Utilisation selon la revendication 3, dans laquelle la composition ne contient pas de zinc.
5. Procédé de formation d'une composition d'huile non newtonienne sous la forme d'une graisse comprenant un sulfonate de calcium surbasé et une huile de base renouvelable, (1) chauffage de sulfonate de calcium surbasé, d'hydroxyde de calcium et d'un agent de conversion, comprenant une huile de base renouvelable ayant un KV100 dans la plage de 3,0 à 10,0 cSt ; un point d'écoulement dans la plage de -20 à -55 °C; une relation de Noack et CCS à -35 °C telle que Noack est compris entre 2 750 (CCS à -35 °C) $(-0,8) \pm 2$; (2) mise en réaction dudit produit de l'étape 1 avec des composants comprenant un composé d'acide borique pour développer les propriétés analogues à une graisse, dans lequel l'huile de base renouvelable est la seule huile de base utilisée dans le procédé.
6. Procédé selon la revendication 5, dans lequel la réaction se déroule en présence d'acide 12-hydroxystéarique.
7. Composition de graisse comprenant une huile de base renouvelable et un épaississant, dans laquelle l'huile de base renouvelable comprend un KV100 dans la plage de 3,0 à 10,0 cSt ; un point d'écoulement dans la plage de -20 à -55 °C; une relation de Noack et CCS à -35 °C telle que Noack est compris entre 2 750 (CCS à -35 °C) $(-0,8) \pm 2$; et l'épaississant est choisi dans le groupe constitué par le lithium, un complexe d'aluminium, une base d'argile et une polyurée ; et dans lequel l'huile de base renouvelable est la seule huile de base présente dans la composition.
8. Composition selon la revendication 7 dans laquelle l'huile de base renouvelable comprend les paramètres RMN

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suivants : (a) BP/BI des mélanges hydrocarbonés sont dans la plage de $\geq -0,6037$ avec ramification d'alkyle interne par molécule + 2,0 et (b) les méthyles 5+ des mélanges d'hydrocarbure sont en moyenne de 0,3 à 1,5 par molécule.

5 9. Composition selon la revendication 8, dans laquelle la composition ne contient pas d'additif de performance supplémentaire.

10 10. Composition selon la revendication 8, dans laquelle la composition contient des additifs de performance supplémentaires.

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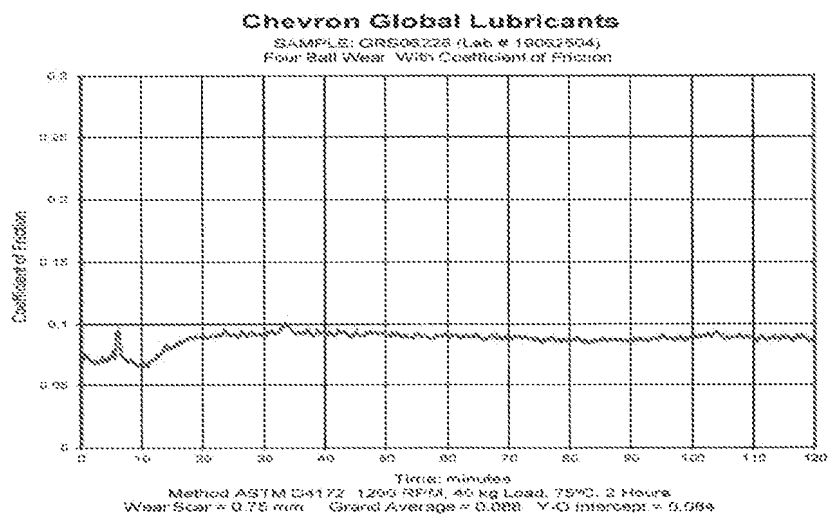
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FIG. 1

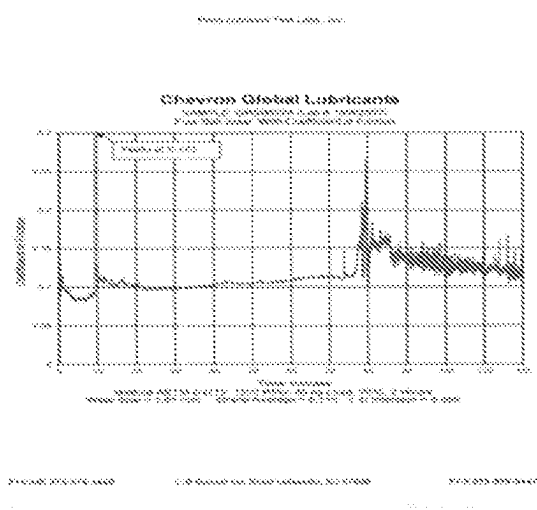


FIG. 2

REFERENCES CITED IN THE DESCRIPTION

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