RESEARCH ARTICLE

Preparation and Evaluation of Sulfonate Polyethylene Glycol Borate Ester as a Modifier of Functional Properties of Complex Petroleum Lithium Grease

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The goal of our study is the synthesis and evaluation of new additives to increase the dropping point and improving the extreme pressure (EP) properties of prepared Lithium 12-hydroxy stearate grease samples. High dropping point, extreme pressure properties (EP) and low oil separation are often the most important factors. The target additive, polyethylene glycol borate esters were synthesized via the stoichiometric reaction of poly ethylene glycol 400 and boric acid to give polyethylene glycol borate ester (PB) which reacted with the linear alkyl benzene sulfonate (1:1) in the presence of toluene as azeotrope to give the target sulfonate ester (SPB). The structure of the new synthesized polyethylene glycol borate esters (PB) and (SPB) were confirmed by using FT-IR. High performance liquid chromatography technique was used to confirm the purity and reactivity of the borate ester (PB) as a precursor for the synthesis of the target additive (SPB). The two new synthesized additives showed the ability to improve some properties of the prepared grease samples such as grease consistency ASTM D-217, dropping point ASTM D-2265 and oil separation ASTM D-6184. Four–Ball test ASTM D-2783 showed better capacities of (SPB) may due to the presence of the sulfonate group.

Introduction

Polyethylene glycol borate ester have attracted our attention, because they are non-toxic and exhibit pleasant odor, good oxidation inhibition ability, outstanding friction-reducing and anti-wear ability, high load-carrying capacity and a certain degree of biodegradability [**1-3**]. On the other hand, poly ethylene glycol (PEG) and its derivatives are used in different industrial applications [**4-6**] resulting from their low cost and useful physical properties, such as solubility in aqueous and organic solvents. Furthermore, boric (III) acid is of interest because it is including many acceptor oxygen atoms which can form proton channels, where in such chain's oxygen atoms are present the hydrogen-bonded channels are formed which show large proton polarizability [**7-10**]. Greases are complex systems consisting of base oils and thickeners based on soaps or other organic or inorganic substances. Grease, which plays key roles in reducing and/or preventing friction and wear of mechanical systems in various industrial fields, and the performance of various advanced lubricants highly, depends on functional additives [**11**], other ingredients imparting general properties may be included as additives for extreme pressure, anti-wear, rust prevention, etc. Greases can be classified according to either, base oil, thickener and applications [**12,13**]. Complex soap is defined by the NLGI as: "a soap wherein the soap crystal or fiber is formed usually by co-crystallization of two or more compounds; normal soap and complexing agent. In further explanation, the complexing agent is said to be the ability to change in grease essential quality, usually recognized by an increase in dropping point [**14**]. While co-crystallization requires the two compounds to be present during the high temperature heating phase of the grease cooking process and for them to crystallize in close contact together during a transition from fluid to semi-solid form, it's thought that this is not the only method of complex formation [**15**]. It is important to know that the NLGI grades are allocated from the P60 worked penetration figures.

The NLGI has classified greases, **Table 1**, according to consistency as measured by penetration [**15**].

Table 1. Penetration of classified greases according to NLGI.

NLGI number	Penetration range
000	445 - 475
00	400 - 430
0	355 - 385
1	310 - 340
2	265 - 295
3	220 - 250
4	175 - 205
5	130 - 160
6	85 - 115

It has been concluded that during the "complexing" process there is not a formation of new chemical compounds, but rather strong adsorption interactions develop between the soap and complexing agent during preparation of the grease [**16**]. Complex greases are commonly specified for use in high-temperature industrial applications, the usual thickener system in lithium complex grease consists of the Lithium salt of 12-hydroxystearic acid plus a complexing agent. The higher value of high-temperature application range is balanced against higher complex soap content, increased batch manufacturing cycle time and reduced pumpability compared to simple lithium grease [**17,18**]. Lithium complex grease is marketed as a superior of performance versus lithium simple soap grease. This superiority is a result of increased operating temperature limits, which can be more than 100°F (56°C) higher than the simple lithium soap [**19**]. From this point of view, it is important to develop high-performance lubricant additives. We also demonstrate here the utility of high-performance liquid chromatography (HPLC) for monitoring the formation, purity, and particularly the integrity of PEG derivative (BP), since polymer degradation was unexpectedly found in some instances under relatively mild reaction conditions. Also, it has been reported that [**20**], the use of boron Esters improve the high temperature capability of Lithium 12-Hydroxystearate Soap. The main idea of this work depends on the synthesis and identification of new borate ester additives which improve the properties of Lithium 12-Hydroxystearate grease such as dropping point, antioxidant, anti-wear, hardness or fluidity.

Consistency

The consistency (hardness) of a grease is defined as the depth (in tenths of a millimeter) to which a standard testing cone penetrates a grease sample under prescribed conditions.

Grease additives

Teflon is added to some greases to improve their lubricating properties [**21**]. Gear greases consist of rosin oil, thickened with lime and mixed with mineral oil, with some percentage of water [**22**]. Under high pressure or shock loading, normal grease can be compressed to the extent that the greased parts come into physical contact, causing friction and wear [**23**]. Extreme pressure grease, {grease labeled "EP"} which contains solid lubricants, usually graphite and/or molybdenum disulfide provide protection under heavy loadings, where the solid lubricants bond to the surface of the metal and prevent metal-to-metal contact and the resulting friction and wear when the lubricant film gets too thin. Coppaslip is the registered trademark of one such grease produced by Molyslip Atlantic Ltd., and has become a generic term (often misspelled as "copper slip" or "coppaslip") for anti-seize lubricants which contain copper [**24,25**]. Grease is expected to be tolerate moisture, compatible with seals, reduce friction and wear, provide corrosion protection, resist leakage, dripping and throw-off, maintain mobility under conditions of application. **Table 2** shows the classification of greases according to these properties.

Table 2. Classification of greases.

Classification Item		
Base oil	**Thickener type**	**Application**
A- **Mineral oil:** as, Paraffinic Naphthenic	A- **Organic thickener:** a. Soap type: i. Simple grease ii. Complex grease b. Non-soap type: as i.Poly urea ii.Calcium sulfonate	Bearing grease Coating grease High Temperature / Extreme Pressure Grease Conveyors
B- **Synthetic oil**: as, Poly alpha olefin Ester oil Silicon oil	B- **Inorganic thickener:** as i. Bentonite (Clay) ii. Fumed silica	Chain Chassis, etc.

Materials and methods

General

Boric acid, polyethylene glycol and alkyl benzene sulfonic acid and other reagents were purchased from Sigma Chemical Co. FT-IR spectra was run on a ALPHA FT-IR Spectrometer from Bruker USA (Misr Petroleum Company, Cairo, Egypt) and expressed in wave number cm^{-1}. HPLC was carried out on an Agilent 1200 Infinity Series spectrophotometer (Misr Petroleum Company, Cairo, Egypt). Grease consistency ASTM D-217 and Dropping point test ASTM D-2265 were performed at Misr Petroleum Company, Alexandria, Egypt. Antioxidant activity, four–Ball test ASTM D-2783, Oil separation: ASTM D-6184 were performed at Misr Petroleum Company, Cairo, Egypt. Purity of both starting materials as well as the reaction products were checked by TLC using Macherey-Nagel Alugram SilG/UV254 silica gel plates.

Synthesis of the target additives

Synthesis of polyethylene glycol borate ester (PB)

In a 250-ml two necked round bottomed flask connected through a separator attachment ("trap") for separating water to a reflux condenser; thermometer is inserted into the other neck a mixture of boric acid and polyethylene glycol 400 (1:1) was heated under reflux for about 5 hours with stirring by mechanical stirrer (150 rpm) at about 180ºC, 100 ml of toluene was used as azeotrope. The reaction mixture was extracted with petroleum ether, distilled under reduced pressure, polyethylene glycol borate ester was isolated as yellow viscous liquid 98 % yield.

This method was performed as normal esterification conditions [**26-28**]. **Table 1** represents the conditions of condensation of boric acid & polyethylene glycol 400 to obtain polyethylene glycol borate ester.

Synthesis of sulfonyl polyethylene glycol borate ester (SPB)

In a 250-ml two necked round bottomed flask connected through a separator attachment ("trap") for separating water to a reflux condenser; thermometer is inserted into the other neck A mixture of linear alkyl benzene sulfonic acid and polyethylene glycol borate ester (PB) (1:1) was heated under reflux for about 6 hours with stirring, The reaction mixture was extracted with petroleum ether, distilled under reduced pressure, sulfonyl polyethylene glycol borate ester was isolated as slight brown paste 80%.

Preparation of Li-12 hydroxy stearate grease

Reagents

N-13 Naphthenic oil (33.45%), 12-hyroxystearic acid (5.25%), Hydrogenated castor oil (3.37%), Lithium hydroxide monohydrate (1.27%), P-30 (Bright Stock) Paraffinic oil (56.66%).

Grease preparation conditions

The experimental work was hold in a simple system consists of glass beaker of one liter, with agitator motor and an electrical heater with temperature control.

N-13 Naphthenic oil was weighed in the beaker and heated at 120 ºC, and the fatty materials 12-hyroxystearic acid & hydrogenated castor oil was added gradually with moderate speed, with the reaction temperature 212-215ºC. A suspension of $LiOH.H_2O$ in oil was added gradually to the fatty material, when all $LiOH.H_2O$ was completely added, raise the temperature to 230ºC for 5 minutes, cool the reaction temperature slowly (1ºC each 1min.) to 180ºC. Start adding paraffinic oil drop by drop every 10 minutes, until the grease is completely formed add the rest oil for cooling and adjusting the grade (consistency) of the grease.

Characterization tools

IR spectra

The infra-red spectra [**29**] of the synthesized borate esters were carried out to detect the esterification process. Fig. 1 and Fig. 2 represent the IR Spectra of the synthesized borate esters (PB & PBS respectively).

HPLC analysis

The high-performance liquid chromatography method [**30**] was used to confirm the purity and reactivity of the borate ester (PB). The HPLC assay was conducted on an Agilent 1200 series liquid chromatograph (Agilent, USA). The stationary phase was X Bridge C18 with 5 m particle diameter (Waters, Ireland). The column dimensions were 250 × 3.0 mm. The mobile phase flow rate was 0.5 mL/min. The mobile phase consisted of 0.1% formic acid in water as aqueous component and 100% acetonitrile as organic modifier. A generic gradient LC method with a run time of 30 min was developed for the purity analysis of PEG 400 oligomers. The column and autosampler were maintained at 40ºC and 4ºC, respectively. The ELSD was operated with typical settings as follows: evaporation temperature, 75ºC; nebulizer temperature, 80ºC; gas, 1.65 SLM.

Preparation of calibration standard and sample

Master stock solution of PB (1 mg/mL) was prepared in DMSO. Working standard solutions of PEG 400 were prepared by serial diluting from master stock (PEG 400 provided by supplier with density of 1.126 g/mL was used as master stock) with acetonitrile: DMSO: water (2:2:1).

A 100 µL aliquot of sample (PB) was pipette in to a 96-well polypropylene plate and extracted with 200 µL of acetonitrile containing internal standard. Peak areas for all components were automatically integrated.

Evaluation tests of additives

Grease consistency ASTM D-217 is a Standard test method for cone penetration of lubricating grease, to measure the hardness or fluidity of grease. The penetration test begins with the grease at 25°C, plus or minus 1 degree Celsius, being levelled into a cup. The sample size and cone weight for this test are determined [**15,16**]. Using a penetrometer, a cone is dropped into the cup for 5 seconds, creating a hole in the grease, the depth in a tenth of a millimeter of this hole was determined. This value is known as the P0 or unworked penetration. The grease is then sheared (or worked) using a mechanical hand operated device through 60 double strokes, the cone test was repeated to determine the P60 value.

Dropping point is the temperature at which the thickener releases the oil and get separate of it, so this test is very important in detecting the grease application temperature.

ASTM D-2265 this standard test was carried out to the prepared grease sample and the prepared grease sample with the synthesized polyethylene glycol borate esters with different doses to evaluate the effect of additive on the grease.

Four–Ball test ASTM D-2783 is a standard test for the evaluation of the anti-wear characteristics of lubricant and its ability to lubricate under extreme pressures and high loads. This test was carried out at the optimum ratio of synthesized sulfonate polyethylene glycol borate ester blend with the prepared grease sample to evaluate the Anti-wear function (scare diameter) and Extreme Pressure effect (Welding load). Before each test, the specimens were cleaned in petroleum ether, and then dried. A microscope was used to determine the wear scar diameters (WSD) of the three lower balls with an accuracy of 0.01 mm, and the friction coefficient was recorded automatically. The welding load should be as high as possible, while the scar diameter should be as low as possible.

Oil separation: ASTM D-6184 this standard test method measures the amount of oil separated from grease under test conditions, the lower such number is, the better is the grease.

Results and discussion

Objective

1. Preparation of polyethylene glycol borate esters (PB) & SPB) as grease additives.
2. Prepare Li-12 hydroxy stearate grease to evaluate the synthesized additives.

Features

High dropping points and good E.P properties

In our study, we compare the characteristics and effect of synthesized borate esters additives on Lithium 12-hydroxystearate grease comparing it with lithium complex grease based on azelaic acid as complexing agent. The synthesized borate ester additives[2] have the effect of raising the dropping point of grease containing 12-hydroxystearic acid-based soap. The difference between this technique and the use of conventional complexing agents is that the borate esters do not need to be present in the cooking stage; instead borate ester additive is added along with other performance enhancing additives, such as extreme pressure, anti-wear, antioxidant and corrosion inhibitors, near the end of the manufacturing cycle, just prior to packaging at the finishing stage of a typical batch manufacturing process. The explanation of the mechanism for the improvement of dropping point to the level of complex soaps theorized that a stable, coordinated compound is formed by electron sharing between the boron atom of the borate ester compound and the hydroxyl group of the 12-hydroxystearic acid soap.

$$M\text{-}OOC\text{-}(CH_2)_5\text{-}CH(\ddot{O}H)\text{-}(CH_2)_{10}\text{-}CH_3 + \ddot{B}\text{-}(OR)_3$$

where **M** is metal atom may be Lithium (Li^+) or Sodium (Na^{+}) or Calcium (Ca^{+2}) or Aluminum (Al^{+3}) and **R** is alkyl group from C_4 to C_{16}.

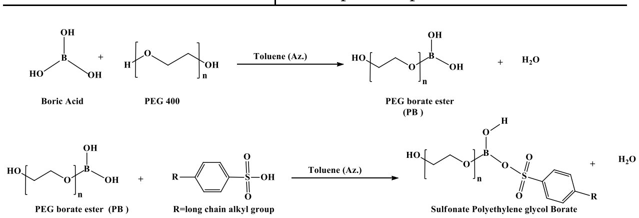
In doing so, the interactions between soap and complexing agent produce a change in the high-temperature properties of the finished grease, allowing it to maintain its consistency far beyond the melt point of simple soap grease.

Preparation of polyethylene glycol borate ester additives

Polyethylene glycol borate ester additive (PB) was synthesized by using boric acid and poly ethylene glycol 400 (1:1), **Table 3** shows the condition of the reaction, polyethylene glycol borate ester in turn reacted with linear alkyl benzene sulfonic acid (1:1) in presence of toluene as azeotropic to give sulfonate Polyethylene glycol borate ester additive (SPB). **Scheme 1** shows the reaction pathway for the synthesis of the two additive esters.

Table 3. The Optimum Conditions for the synthesis of borate esters (PB) & (SPB).

Item	Data
Temp., ºC	Up to 180
Reaction time, hr	5
Stirring rate/rpm	150
Pressure	atmospheric pressure



Scheme 1. Synthesis of sulfonate polyethylene glycol borate ester (SPB).

Structure analysis

The infrared spectra results of PB and PBS (**Fig. 1** and **Fig. 2**), showed that the peak at 3373 cm^{-1} (PB), 3196 cm^{-1} (SPB) are the stretching vibration absorption peak of the unreacted hydroxyl groups, the stretching vibration peaks at 2921.16, 2852.41cm^{-1} (PB), 2510.64, 2358.81, 2259.35 cm^{-1} (SPB) are the C-H bonds (CH_2 & CH- 2 or 3 bands), the B-O (belongs to B–O bonds of trigonal BO_3 units)asymmetric stretching vibration absorption peak is at 1415.81 cm^{-1} (PB), 1415.60 cm^{-1} (SBP), also the infrared spectrum of (SBP) showed two absorption characteristic peaks appeared at 1192 cm^{-1} corresponding to sulfonyl group and the peak at 882.19 cm^{-1} corresponding to S-OR sulfonate ester.

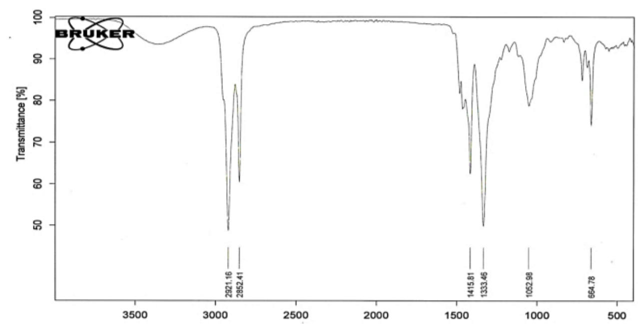


Fig. 1. IR Spectrum of polyethylene glycol borate ester (PB).

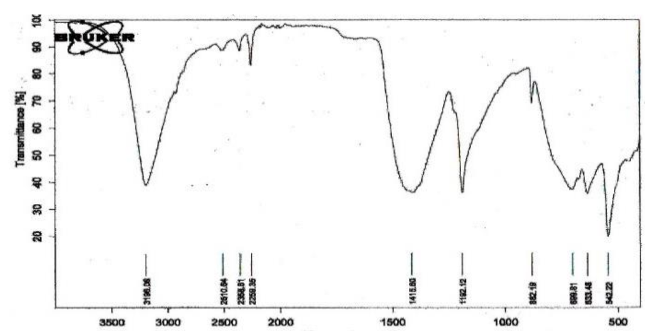


Fig. 2. IR Spectrum of sulfonate polyethylene glycol borate ester (SPB).

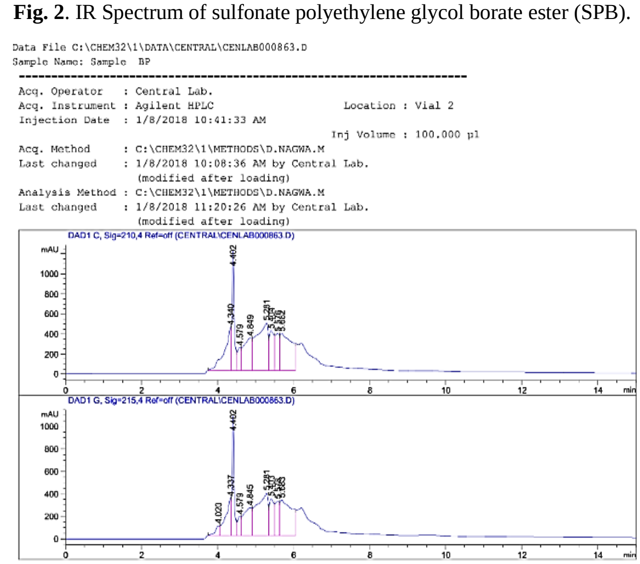


Fig. 3. HPLC diagram of polyethylene glycol borate ester (PB).

The high-performance liquid chromatography of PB as a precursor for the synthesis of the target ester results are shown in **Fig. 3.** Which showed its purity and stability which meet the requirement of lubricating oil additive.

Table 4. Evaluation tests of the prepared grease and the grease samples with sulfonate polyethylene glycol borate ester additive of different doses.

Grease type	Grease A	Grease B				Grease C			
Description	Lithium 12- hydroxy stearate	Lithium 12- hydroxy stearate + P.E.G borate ester (PB)				Lithium 12- hydroxy stearate +Sulfonate P.E.G borate ester (SPB)			
Additive dose, %	0	1	2	3	4	1	2	3	4
Dropping point, ºC	200	230	285	320	327	225	280	330	330

Evaluation of additives application

The effect of the synthesized additives on improvement the dropping point of the grease at different doses are illustrated in **Tables (4)**, comparing it with synthesized grease without any additive (Grease A). The results depicted concluded that with increasing the concentration of the additive (PB) & (SPB) the dropping points increases, and the dropping point at 3% showed the favorable concentration for both additives **Fig. 4** & **Fig. 5.**

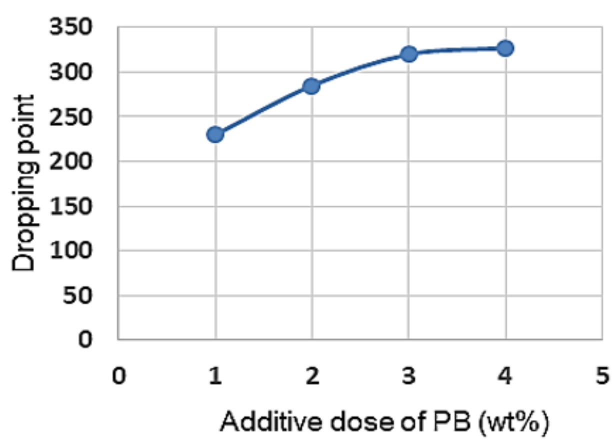


Fig. 4. Effect of polyethylene glycol borate ester on dropping point of Lithium12- hydroxy stearate.

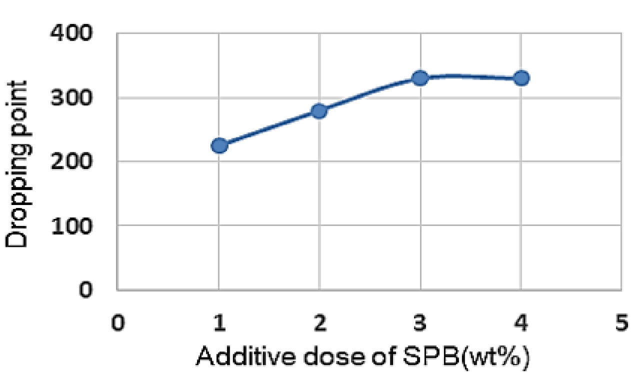


Fig. 5. Effect of sulfonyl polyethylene glycol borate ester on dropping point of Lithium12- hydroxy stearate.

On the other hand, the dropping points at this concentration still the influencing factor even after a long period of time, where the dropping point at this concentration for (PB) & (SPB) after fourteen months, the value becomes 310&320 respectively. Other characteristic functional properties for the title additive ester (SPB) at 3% concentration such as NLGI, grease consistency, extreme pressure properties, welding load and oil separation were estimated comparing it with simple and complex grease as shown in **Table 5.**

Table 5. Evaluation tests of the prepared grease and the grease sample with borate ester additive and complex grease.

Grease type	Grease A	Grease B	Grease C	Grease D
Description	Base Lithium 12- hydroxy stearate	Base Lithium 12- hydroxy stearate+3% P.E.G borate ester (PB)	Base Lithium 12-hydroxy stearate+3% P.E.G sulfonate borate ester (SPB)	Lithium 12- hydroxy stearate: Azelerate complex grease
NLGI, grease consistency	2	2	2	2
Dropping point, ºC	200	320	330	275
E.P. properties, welding load, Kg	150	160	250	200
Oil separation, % max.	0.5	0.2	0.2	0.3

Benefits

- Applicable in other 12-hydroxystearate grease such as, Sodium and Calcium.
- Easy to manufacture.
- Low cost comparing with imported complex grease.
- Wide applicable temperature range.

Conclusion

The synthesized polyethylene glycol (400) borate esters (PB) was used for the improvement of high temperature properties of Li 12-hydroxy stearate grease, besides P.E.G sulfonate borate ester (SPB) itself was used not only also for the improvement of high temperature properties of Li 12-hydroxy stearate grease but also for the improvement of its extreme pressure properties. As they can be used in different doses, where the percentage of additive depends on the different conditions of grease used. For the grease manufacturer, this adds significant flexibility as well as cost savings over conventional methods of obtaining high-temperature soaps. Further studies to explore the antioxidant and anti-rust of the two synthesized additives are currently underway in our laboratory.

Keywords

Polyethylene glycol, grease, dropping point, extreme pressure (EP), additive, borate-esters.

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