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BASE OIL BLENDS - A GREAT OPPORTUNITY FOR GREASE FORMULATORS

Abstract

Mineral oils used by grease manufacturers can be divided into two major groups, naphthenic oils and paraffinic oils. These categories of base oils have their own advantages and disadvantages depending on the applications and conditions. The most important advantages of the naphthenic oils over the paraffinic oils, with the same viscosity and similar aromatic content, are better low-temperature flowability and better solvency. The contribution of having base oils with good solvency towards the thickener is that less thickener is needed to obtain a certain consistency of the finished product. For instance, a typical NLGI grade 2 lithium based grease, based on a solvent neutral 500, group one (Gr I) oil, may contain 9-14 wt% thickener; while 6-8% thickener is required for an equivalent viscosity oil of naphthenic nature. Using blends of naphthenic oil and paraffinic group one oil have successfully been used during the past decade in India. However, the author believes that the surplus of paraffinic Gr II and Gr III base oils is one among a number of market trends that brings some great opportunities to the grease formulators, if they look for blends where naphthenic oils are regarded as part of the solution for a sustainable formulation. A number of key parameters important for the performance of the finished product could be obtained within reasonable cost.

The purpose of this work was to compare "side by side" three base oil blends where paraffinic Gr I, Gr II and Gr III, in combination with naphthenic, were used for preparation of lubricating greases. Since the target viscosity was 150 mm²/s at 40°C, a naphthenic oil was used in order to reach this viscosity.

The overall results obtained, reveal some interesting aspects of the use of Gr II as a substitute to Gr I for preparation of greases. The outcome of this work emphasizes that blends should be regarded as a great opportunity for grease formulators who are looking for some further development of their current formulations and furthermore, the lubricating grease based on the blend of paraffinic Gr II and naphthenic oil performs better than others.

Key words: *traction coefficient; low temperature flowability; oxidation stability; elastomer compatibility; thickener content; naphthenic oil; paraffinic oil; lithium based grease*

Preparation and characterization of the base oils

The target for this study was to compare different oil mixtures, where the main focus was on the possible synergies of the blends between paraffinic oils and naphthenic oil. For this reason, three base oil blends have been prepared by using three paraffinic base oils, representing Gr I, Gr II and Gr III, with one naphthenic oil. Table 1 displays the typical characteristics of the three paraffinic base oils and the naphthenic oil.

Table 1: Typical characteristics of the paraffinic base oils.

| Properties | Unit | Method (ASTM) | Gr I | Gr II | Gr III | Gr V |
|---------------------|--------------------|---------------|------------|------------|------------|------------|
| Type of oil | | | Paraffinic | Paraffinic | Paraffinic | Naphthenic |
| Viscosity at 40 °C | mm ² /s | D 445 | 90 | 110 | 49 | 440 |
| Viscosity at 100 °C | mm ² /s | D 445 | 10.1 | 11.9 | 7.9 | 19.9 |
| Viscosity index | - | D 2270 | 95 | 97 | 130 | 19 |
| Density at 15 °C | kg/m ³ | D 4052 | 888 | 875 | 820 | 931 |
| Flash point | °C | D 93A | 260 | 269 | 261 | 221 |
| Pour point | °C | D 97 | -12 | -15 | -12 | -12 |
| Aniline point | °C | D 611 | 99 | 124 | 117 | 88 |

As it can be seen in Table 1, the paraffinic oils had different viscosities at 40 °C; subsequently different wt.% of the naphthenic oil was used to obtain 150 mm²/s in viscosity at 40 °C. The characterizations of the base oil blends cover a wide range of tests. Table 2 shows the characteristics of the three base oil blends that have been used for preparation of greases in this study.

Table 2: Characteristics of the base oil blends

| Properties | Unit | Method (ASTM) | Blend A | Blend B | Blend C |
|---------------------------|--------------------|---------------|---------|---------|---------|
| Viscosity at 40 °C | mm ² /s | D 445 | 147 | 149 | 148 |
| Viscosity at 100 °C | mm ² /s | D 445 | 12.4 | 13.4 | 12.8 |
| Viscosity index | - | D 2270 | 66 | 82 | 73 |
| Density at 15 °C | kg/m ³ | D 4052 | 903.2 | 888.8 | 899.4 |
| Refractive index at 20 °C | | D 1747 | 1.496 | 1.487 | 1.495 |
| Flash point | °C | D 93A | 223 | 239 | 235 |
| Pour point | °C | D 97 | -21 | -24 | -27 |
| Aniline point | °C | D 611 | 99.8 | 116.0 | 104.3 |
| Colour | | D 1500 | 4.2 | 3.2 | 4.8 |
| Total acid number | mg KOH/g | D 974 | 0.017 | 0.012 | 0.021 |
| Copper strip test | | D 130 | 1a | 1a | 1a |
| Carbon type composition | wt.% | D 2140 | | | |
| C _A | | | 9.0 | 3.2 | 7.5 |
| C _P | | | 58.6 | 64.6 | 66.4 |
| C _N | | | 32.1 | 32.4 | 21.6 |

Table 2 reveals that the viscosity of the blends at 100 °C varies within 1 mm²/s, which is close enough to be regarded as equivalent to each other. Furthermore, the main impact of the naphthenic oil on the blends can be noted in the parameters such as aniline point.

Tribological measurements of the base oil blends

A mini traction machine (MTM2, PCS Instruments) has been used to measure the frictional properties of the base oil blends in a mixed rolling/sliding contact. A load is applied to a steel ball, and with a force transducer the frictional force is measured as the ball is in contact with a steel disc. The Slide to Roll Ratio (SRR) is defined as the percentage ratio between the sliding speed (difference between the ball speed and the disc speed) and the mean speed. Consequently, 0 % is pure rolling and 200 % is pure sliding. Furthermore, by measuring the traction (friction) as a function of the mean speed at constant SRR, a Stribeck curve can be obtained.

In this study, the friction measurements were performed in a steel/steel contact at 820 MPa and a steel ball with a diameter of 19.05 mm (3/4") was used. Both the steel ball and the steel disc were made of AISI 52100 steel and had a smooth finish with a roughness less than 0.01 µm (Ra). Four consecutive runs were measured, and the mean value of the measurements is displayed. Two tests for each base oil blend were performed and very good agreement was observed for the tests. The temperature was kept constant at 40 °C for all MTM measurements. A Stribeck curve was obtained by measuring the Traction coefficient of the blends as a function of speed at constant SRR (50%), see Figure 1. This Figure indicates a slightly lower traction coefficient for blend B.

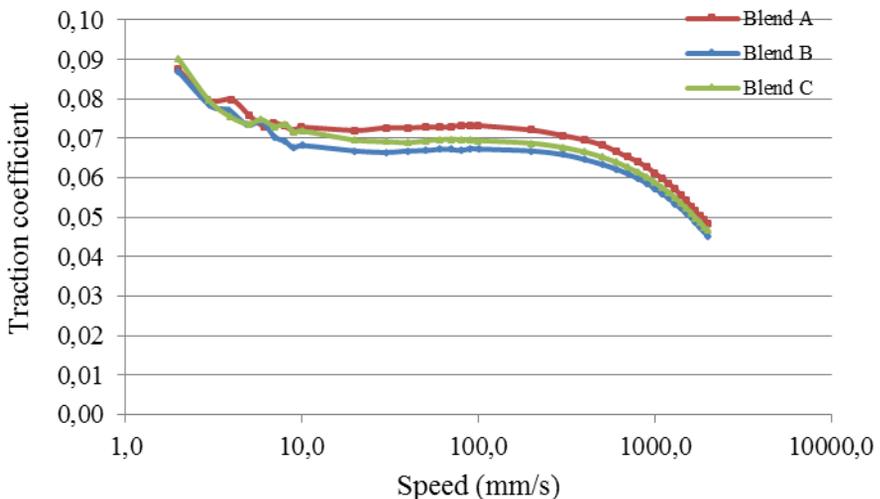


Figure 1: Traction coefficient as a function of speed, measured at 40 °C

Electrical Contact Resistance (ERC)

ERC describes the degree of separation between the ball and the disc in a MTM. Hence, when they are fully separated from each other the voltage at the disc will be the same as the applied one (15 V) and if they are fully separated (100%) e.g. by base oil, then the balance resistor will be 0 V.

Based on the measured ECR, at different speeds, for the three blends, see Figure 2, a better performance for the Blend B at lower speeds can be observed. This measurement reveals that the film thickness of Blend A and C are reduced significantly at lower speeds. However, based on these few measurements, it is not understood why Blend B contributes to more stable film thickness regardless the speed.

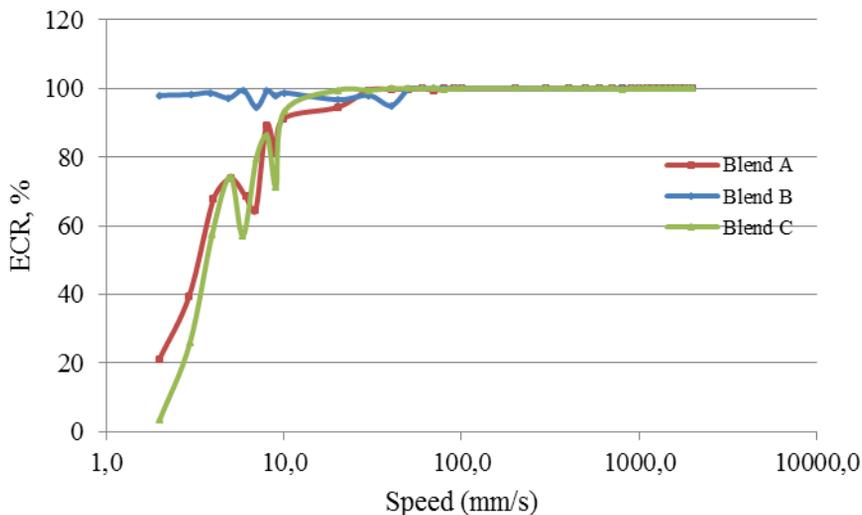


Figure 2: Effect of speed on ECR measurements

Preparation and characterization of the greases

The three base oil blends (A, B and C) have been used for preparation of lithium based greases in an open kettle where lithium hydroxide was reacted with 12-hydroxy stearic acid. Greases A, B and C were made with base oil blends A, B and C respectively. Notable that no additive was used in these greases. In order to make the comparison easier, each blend was used during the entire production of the grease, meaning a blend was used in both cooking and cooling stages.

Based on the obtained results shown in Table 4, some conclusions may be drawn such as:

- Low soap content in Grease C was needed to achieve the penetration target, probably due to a higher wt.% of naphthenic oil used in this blend.
- Good shear stability was achieved for all three greases were obtained.

- The slightly higher wear scar resulted by Grease C probably can be explained by its lower thickener content. The measured wear scars for all three greases are in-line within the expected wear scar for neat lithium based grease.

Table 3: The characteristics of the greases

| Characteristics of the greases | Unit | Method ASTM | Grease A | Grease B | Grease C |
|---|------------------|-------------|------------------|------------------|------------------|
| Base oil type (naphthenic+paraffinic) | | | Blend A | Blend B | Blend C |
| Penetration (0) | mm ⁻¹ | D 217 | 281 | 289 | 283 |
| Penetration (60 str) | mm ⁻¹ | D 217 | 280 | 287 | 284 |
| Penetration (10 ⁵ str) | mm ⁻¹ | D 217 | 296 | 293 | 298 |
| Difference in penetration after 10 ⁵ str | | | +16 | +6 | +12 |
| Dropping point | °C | IP 396 | 202 | 200 | 203 |
| Soap content | wt. % | | 8.23 | 8.83 | 6.39 |
| Wear scar (400 N/60 min) | mm | DIN 51350:5 | 0.86 | 0.89 | 1.00 |
| Cu-corrosion | | D 4048 | 1b | 1b | 1b |
| Texture | | | Smooth & Buttery | Smooth & Buttery | Smooth & Buttery |

Elastomer compatibility

Chloroprene rubber is one of the most common elastomers used in constant velocity joints application. Hence, the rubber interactions with the lithium grease, based on the three blends, were conducted through total immersion of the rubber sample in the greases. The test duration was 168 hours at 100 °C.

The change in hardness and weight of the chloroprene rubber samples was measured. Hardness is a measurement of a rubber's ability to resist penetration of a specified metal rod with a specifically shaped tip or end. In order to measure the change in hardness of the rubber, the IRHD (International Rubber Hardness Degrees) method was used.

Previous publications have shown that the aniline point of the oil has significant influence on the interaction between the oil, the corresponding grease and the rubber. For example, in the case of nitrile butadiene rubber, an oil with low aniline point is recommended, while high aniline point is desired for chloroprene rubber. This part of the work confirms that recommendation. Base oil Blend B, used for Grease B, has the highest aniline point (116 °C), and this is reflected in the obtained results, see Figures 3 and 4.

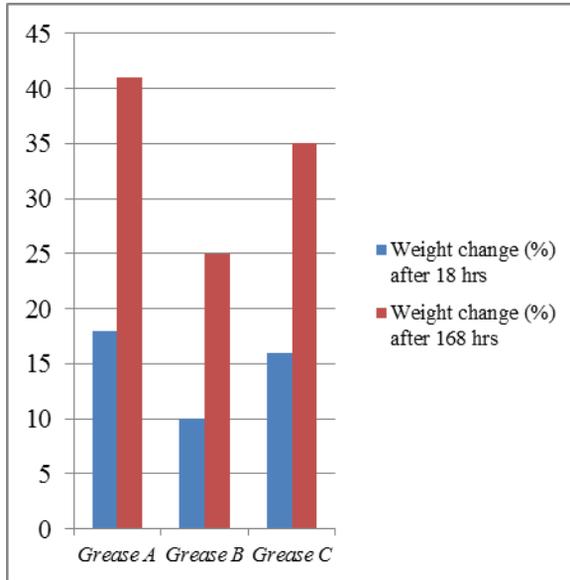


Figure 3: The impact of different greases on C.R. (weight change) after various times at 100 °C.

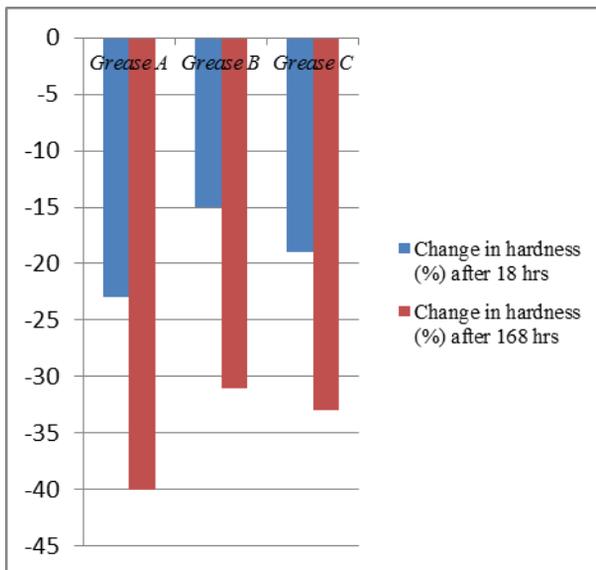


Figure 4: The impact of different greases on C.R. (change in hardness) after various times at 100 °C.

Oxidation stability tests

The standard oxidation stability test according to ASTM D 942, also called rotary bomb test (Norma-Hoffman), has been used. The obtained results, pressure drop, for all three greases were about 2 psi which is regarded to be very good despite of the fact that the greases do not contain any antioxidant. At the last ELGI annual meeting a new oxidation test method for measuring oxidation stability of lubricating grease was introduced by George Dodoes. The suggest method, RSSOT (The Rapid Small Scale Oxidation Test) has been described as a more reliable and faster test for determination of the oxidation stability of lubricating greases. This test is frequently used for measurement of the resistance of various type of fuels (ASTM D 7545). Figure 5 and 6 show the photo of these two test apparatuses respectively.



Figure 5: Rotary bomb test apparatus

According to this method, a breakpoint at with a pressure drop of 10% below max pressure is recorded as the induction time at a constant temperature. Max pressure is the sum of the applied oxygen pressure and the vapour pressure of the sample.

In order to be able to compare the obtained results for these three greases with the published data, it was decided to carry the tests with the following condition: the applied temperature and oxygen pressure has been kept constant at 140 °C and 700 kPa respectively. Table 4 shows the average induction time obtained for the three samples after two runs.

Table 4: Induction time for greases at 140 °C

| Samples | Induction Time (min) | | |
|----------|-----------------------------|-----------------------------|------------|
| | 1 st Measurement | 2 nd Measurement | Mean value |
| Grease A | 499 | 497 | 498 |
| Grease B | 828 | 819 | 824 |
| Grease C | 597 | 573 | 585 |

As it can be seen, RSSOT test reveals a variation between the samples. Grease B (Gr II+naphthenic) > Grease C (Gr III+naphthenic) > Grease A (Gr I+naphthenic). Notable that, according to published data by other authors, fully formulated lithium based grease with similar viscosity has shown to have an induction time of less than 200 min at the same condition.

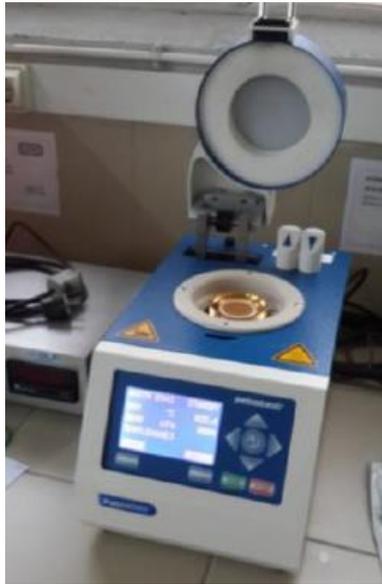


Figure 6: RSSOT apparatus

Flow pressure measurements

In order to simulate the pumpability of the greases, the flow pressure (FP) of the greases at two different temperatures (-20 °C and -35 °C) have been measured, according to DIN 51805. The test setup consists of a conical nozzle that is filled with grease. The nozzle is placed in the measuring instrument, equilibrated for 2-3 hours at a desired temperature, and then an increasing pressure is applied to the nozzle. The threshold pressure at which grease starts to flow through the nozzle is recorded.

Table 5: The impact of temperature on the base oil blends (PP) and the corresponded greases (FP)

| Characteristics | Pour point of the base oil Blends (°C) | FP (mbar), at -20 °C | FP (mbar), at -35 °C |
|-----------------|--|----------------------|----------------------|
| Grease A | -21 | 350 | 1100 |
| Grease B | -24 | 300 | 700 |
| Grease C | -27 | 200 | 525 |

Flow pressure measurements show very good low temperature properties for the greases. However, Grease C (Gr III+naphthenic oil) shows superior low temperature behavior, followed by Grease B (Gr II+naphthenic oil), when compared with Grease A (Gr I+naphthenic).

If the flow pressure for the greases measured at -20 °C are compared with each other it will be found that the differences cannot be explained by the pour point of the base oil blends which is quite interesting. Therefore, in-line with previous finding, using pour point of the base oil in order to predict the flowability of a lubricant could be regarded as a misinterpretation.

Summary

Three base oil blends have been prepared by using a naphthenic oil and three paraffinic oils, one from each group (Gr I, Gr II and Gr III). The characterization of the blends emphasizes that all three blends have similar properties. However, the thermal stability test which is in fact a static test, and MTM measurements, indicate slightly better performance for Blend B, which contains paraffinic Gr II oil.

The three lithium based greases, which are corresponding with the three base oil blends, show good performance in a number of tests. In the rubber compatibility test, Blend B shows less impact, and therefore should be regarded as a better candidate. Regarding the low temperature behavior of the greases, Grease C seems to be a better product.

This study emphasizes the potential of taking advantage of the ongoing fundamental change within the base oil industry, which has created, and will continue to create, new opportunities to upgrade the current (old) grease formulations, by using blends of paraffinic oils, preferably Gr II, and naphthenic oil.

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References

- [1] Salomonsson, L., et al., Oil/thickener interaction of chloroprene and nitrile-butadiene rubber with lubricating greases and base oil. *Lubricating Oil*, 2006: 21(54).
- [2] Serra-Holm V., Development of a novel naphthenic base oil for application in CVJ greases, NLGI 73rd Annual Meeting, October 2006, FL (USA).
- [3] Sullivan, T., Tighter base oil supply expected in Europe. *Lube Report* 2012.
- [4] Fathi-Najafi, M., et al., Low temperature tribology. A study of the influence of base oil characteristics on friction behaviour under low temperature conditions. *EUROGREASE*, 2012.
- [5] Bastardo-Zambrano L. et al., Base oil blends to meet the new demands of the lubricant industry, *Lubes Magazine*, No.115, June 2013.
- [6] Fathi-Najafi M. et al., When the going gets tough... A heavy specialty oil for grease formulations (Part II), NLGI 80th Annual Meeting, June 2013, AZ (USA).
- [7] Dodoes, G. S., A new approach for measuring oxidation stability of lubricating greases, ELGI 26rd Annual Meeting, Dubrovnik (Croatia), 2014.
- [8] <http://www.pcs-instruments.com/mtm/accessories/mtm-cr.shtml#page=page-1>

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