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New perspectives of European oleochemistry
Les nouvelles perspectives de l'oléochimie européenne

New formulations of sunflower based bio-lubricants with high oleic acid content – VOSOLUB project

J.D. Leao[1,⋆], V. Bouillon[2], L. Muntada[3], C. Johnson[4], Paul Wilson[4], O. Vergnes[5], C. Dano[5], A. Igartua[6] and G. Mendoza[6]

[1] ITERG, 11 rue Monge, 33600 Pessac, France
[2] BfB oil research Parc Scientifique Créalys, Rue Phocas Lejeune 10 5032 Gembloux, Belgium
[3] BRUGAROLAS Camino de la Riera, 36-44, Polígono Industrial Cova Solera 08191 Rubí, Spain
[4] RS CLARE Stanhope Street 8-14, L8 5RQ Liverpool, UK
[5] MOTUL Tech, Chemin du Corps de Garde, 77360 Vaires sur Marne, France
[6] IK4-TEKNIKER, Polo Tecnológico de Eibar, c/ Iñaki Goenaga 5, 20600 Eibar, Gipuzkoa, Spain

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Abstract – VOSOLUB project is a demonstration project supported by Executive Agency for Small and Medium-sized Enterprises (EASME) that aims at testing under real operating conditions new formulations of sunflower-based biolubricants with high oleic acid content. These biolubricant formulations (including hydraulic fluids, greases, and neat oil metal-working fluids) will be tested in three European demonstrating sites. Their technical performance will be evaluated and compared to corresponding mineral lubricants ones. In order to cover the demand for the sunflower base oil, a European SMEs network will be established to ensure the supply of the base at a competitive market price. Results presented concerns the base oil quality confirmed to be in accordance with the specification required, in particular on Free Fatty acid content, Phosphorus content, rancimat induction time and oleic acid content (ITERG). The oil characteristics specific for lubricant application analyzed by BfB Oil Research under normalized methods, match with lubricant specifications requirement such as viscosity, cold & hot properties, surface properties, anti-oxidant properties and thermal stability, anti-wear and EP properties, anti-corrosion properties Performance of the new biolubricant have been assessed by formulators and TEKNIKER First results on the use of new lubricant on real condition for rail Grease (produced by RS CLARE and tested with Sheffield Supertram), Hydraulic oil (produced by BRUGAROLAS) and cutting oil (produced by MOTUL TECH and tested with innovative machining, turning) are described.

Keywords: Vegetable oil based lubricant testing / Special fluids / Sustainability / HSE (Health, safety and environment)

Résumé – Nouvelles formulations de biolubrifiants à base d'huile de tournesol à très haute teneur en acide oléique – VOSOLUB Project. VOSOLUB est un projet de démonstration cofinancé par l'Agence Européenne pour les petites et moyennes entreprises (EASME) qui a pour objectif de tester en conditions réelles d'utilisation de nouveaux biolubrifiants sur base d'huile de tournesol à très haute teneur en acide oléique. Ces biolubrifiants (fluides hydrauliques, graisses de lubrification ferroviaire et huiles de coupe) seront testés à travers trois chantier de démonstration au niveau européen. Leurs performances techniques seront évaluées et comparées à celles des lubrifiants sur base minérale. Afin de couvrir la demande en huile de tournesol à très haute teneur en acide oléique, un réseau incluant l'amont et l'aval de la filière sera mis en place pour assurer les approvisionnements à un prix compétitif. Les résultats présentés dans cet article concernent la qualité de l'huile qui est en accord avec les spécifications demandées, en particulier pour l'acidité, la teneur en phosphore, le temps d'induction au rancimat et la teneur en acide oléique (ITERG). Les caractéristiques spécifiques de l'huile pour des applications de lubrification analysées par BfB Oil Research selon des méthodes normalisées, répondent aux spécifications requises pour les lubrifiants telles que la viscosité, les propriétés à froid et à chaud, les propriétés de surface, les propriétés antioxydantes et la stabilité à l'échauffement, les propriétés antiusure et anticorrosive. Les performances de ces nouveaux lubrifiants ont été validées par les formulateurs et TEKNIKER.

Dossier

⋆ Correspondence: jd.leao@iterg.com

Les premiers résultats de l'utilisation de ces biolubrifiants en conditions réelles (graisse de lubrification des rails de chemin de fer produite par RS CLARE et testé avec Sheffield Supertram, huile hydraulique produite par BRUGAROLAS et huile de coupe produite par MOTUL TECH) sont présentés.

Mots clés : Lubrifiant sur base d'huile vegetale / fluides speciaux / durabilite / HSE (Hygiène, Sécurité Environnement)

1 Introduction

In the lubricant sector, like in other sectors, there is a growing need to provide more eco-friendly products such as biolubricants. The VOSOLUB project is a demonstration project that aims at testing under real operating conditions new formulations of sunflower-based biolubricants with high oleic acid content. These biolubricant formulations (including hydraulic fluids, greases, and neat oil metal-working fluids) will be tested in three field tests in real conditions of use. Their technical performance and environmental impacts will be evaluated and compared to corresponding mineral lubricants ones. In order to cover the demand for the sunflower base oil, a European SMEs network will be established to ensure the supply of the base at a competitive market price.

The formulations that will be evaluated have been previously developed in the framework of the FP6 IBIOLAB project using base oils derived from a mild refining process for raw materials developed by the project partner ITERG and from new varieties of vegetable oils with higher oxidative stability. The new process coupled with the use of new varieties of vegetable oil enabled to decrease the overall cost and to increase the technical performance of the bio-lubricants. The developed formulations were very promising some of which could be eco-labelled.

This paper presents analytical results on based oil in relation with lubricant specifications requirement and performance tests results on the three targeted application.

2 Material and methods

2.1 Very high oleic sunflower oil (VHOSO) production and Lubricant formulation

Harvest of sunflower seeds have been done by ARTERRIS, Agricola cooperative from the south of France in late September 2012. Harvest has been done in good condition, impurity level and yields were normal. 25 T of seeds with 87% of oleic acid minimum content have been identified and traceability has been controlled until delivery to crushed plant.

Crushing has been carried out in middle of October 2012 by MEDIACO located in SETE south of France. Crushing has been done in classical conditions and quality of crude oil was in compliance with classical requirements. 15T of crude oil have been produced and sent to PROVENCE HUILES (Vitrolles France) for refining in November 2012.

Refining of crude oil has been done under conditions predefined for a "soft refining". This kind of refining is different from a classical refining (for food application) in which operational conditions are adapted for lubricant application and allow (i) to save energy (due to lowest temperature during deodorization); (ii) to minimize production of by product (deodistillate); and (iii) to minimize loss of natural antioxidant (tocopherol) which have interest for lubricant application.

Process conditions for soft refining used by Provence Huiles were classical neutralization, bleaching 90/100 °C during 30 mn, deodorisation at160 °C during 2 h following by nitrogen inerting before packaging in drums.

Due to industrial constraints (continuous process 24 h/24 h), it was not possible to refine crude oil dedicated to lubrication application in one batch. So 15 tons of "VOSOLUB crude oil" have been injected in on refining run. In order to be sure that the oleic acid content was superior to 87%, monitoring of oleic content have been done during the process for assessment. VHOSO had followed continuously classical neutralised used for sunflower oil. When oil getting out of the neutralizer had 87% of oleic acid content, neutralized oil have been stored in a special tank for feeding the bleacher. The same process has been used for feeding the deodorizer. This procedure that can be reproduced in the future to provide higher quantity of VHOSO allows to guarantee a content of oleic acid superior to 87% that has been confirmed by analytical control on soft refining oil.

2.2 Lubricant formulation and performance test

2.2.1 Cutting fluids performance tests – turning tests on a real CNC machine (Fig. 1)

Tests have been performed in a CNC CMZ lathe TL 15 M 8 (5000 rpm, 14 kW) using a conventional flooding system that impulses the cutting fluid at a conventional pressure with a volume of 40 l/min. Two cutting fluids have been analyzed, the reference mineral (SUPRACO 4018) and the new developed VHOSO based fluid (FIEC 13024). Both cutting oils have been used in pure conditions without emulsifying and have been projected to the cutting zone with and angle of 20 degrees through the conventional piping system.

The material used to perform the machining tests was: Alloy steel AISI4340 (DIN 40NiCrMo7) (quenching and tempering to 31HRc) bars of diameter 110 mm and length 260 mm supplied by IMS group. Concerning the cutting tools, CNMG 120408 LP UE6110 inserts from Mitsubishi with a CVD coated Grade for steel have been used.

The cutting conditions have been selected taking into account mainly some extreme conditions in this interval in order to look for a significant wear during a machining operation of about 20 min and a moderate consumption of machining material.

Fig. 1. Lathe: testing machine.

Another consideration in the final decision of these conditions has been to achieve only flank wear in the cutting edge avoiding crater wear that could result in a catastrophic break of the tool. The test conditions selected have been cutting speed (Vc): 300 m/min, feed rate (F): 0.3 mm/rev, depth of cut (Doc): 0.3 and 0.5 mm.

During machining tests tool wear and workpiece surface qualiy (Ra) have been monitored:

Tool wear. Maximum flank wear is recorded with a contact Microspe Keyence VH-5901 and an optic system with 200× resolution through:

- Progressive observation of the cutting edge. Pictures of edges and flank wear have been taken every 1 or 2 cuts of the cylindrical part.
- Wear measurement in the flank face of the insert considering the tool life criterion:

ISO 3685:1993E standard with a flank (Vb) wear limit of 0.3 mm or

- Machining time of 20 min when the previous threshold is not reached.

Workpiece surface quality. Roughness (Ra) obtained by portable perfilometer Mitutoyo SJ-201 every 1 or 2 cuts.

2.2.2 Hydraulic fluid performance tests

Ball-on disc abrasion tests (rotational motion)

"Ball on disc" tests have been made to study the basic tribological properties of the reference mineral fluid (BESLUX HIDRO HV-46) and new developed bio hydraulic Fluid (BESLUX HIDRO ECO-46) under rotational movement. A standard Falex multispecimen tribometer has been used for that purpose. In this test a ceramic ball rotates against a fixed steel disc. The friction coefficient and wear are recorded during the test. By means of this test, the capacity of the test to avoid the abrasion can be evaluated.

Testing conditions and materials

- Upper specimen: Si_3N_4 balls.
- Lower specimen : Steel disc.
- Initial temperature: room temperature.
- Speed: 3.2 m/s.
- Load: 30 lb.
- Time: 165 min.

FZG Scuffing test (DIN 51354-1/2)

The load capacity of the developed hydraulic fluid has been analyzed with the FZG equipment (see Fig. 2a) according to the DIN 51354-1/2. In this test special gear wheels (Fig. 2b) are run in the lubricant under test, at a constant speed for a fixed time, in a dip lubrication system. Loading of the gear teeth is raised in stages. After load stage 4 the pinion gear teeth flanks are inspected for damage and any changes in tooth appearance are noted.

2.2.3 Grease application performance tests-wheel/rail simulation tests

The simulation wheel/rail tests have been performed in the Twin disc tribometer, using the Twin disc configuration" see Figure 3. It is a test configuration widely used by different research groups in this field, because it is possible to simulate in a simple way the wheel/rail contact. The test consists basically on two small wheels or discs in contact that rotates independently at a fixed angular speed with the defined contact pressure. The test samples have been cut directly from the real wheel and rail.

The testing conditions have been set up, according to the train manufacturer supplied data, trying to simulate the real working conditions.

Testing conditions	Test1	Test
Load	1000 N	2000 N
Pressure	1.3 GPa	1.8 GPa
Rail speed	950 rpm	
Wheel speed	1000 rpm	
Sliding %	5.1%	
Time	8 h	
Number of cycles	480 000	
Contact	3 mm	

The tests have been performed into two steps, during the first 8 h the testing samples (Wheel and rail) have been submitted to a pressure of 1.3 GPa. After evaluation of the wear occurred during the first step, the same wheel and rail samples have been submitted to a second test of 8 h increasing the pressure to 1.8 GPa and maintaining the rest of testing parameters.

3 Results

3.1 Base oil quality

The oil characteristics specific for lubricant application have been analyzed by Bfb (http://www.iespm.com/bfblab/bfblab/index.html) under normalized methods. As illustrated

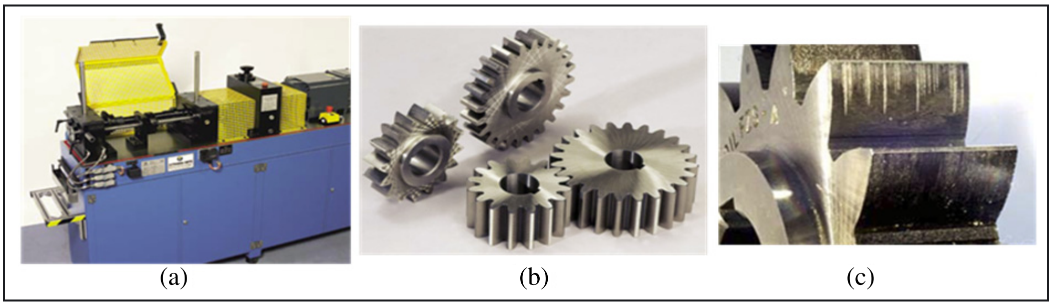

Fig. 2. FZG machine (a), testing specimens (b), example of FZG gear teeth with scuffing marks.

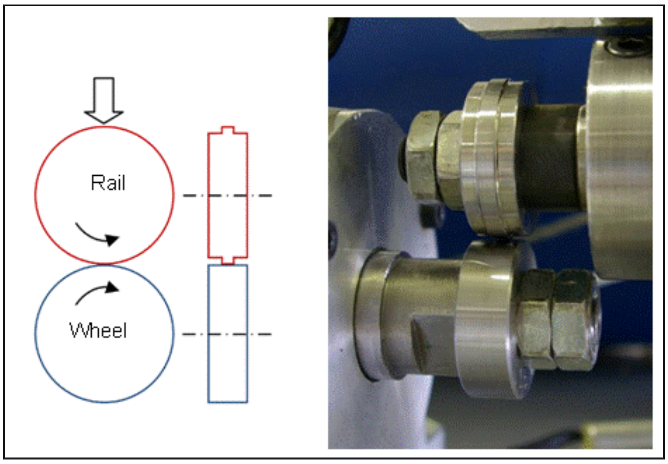

Fig. 3. "Twin disc" configuration.

by Table 1, Results match with lubricant specifications requirement such as viscosity, cold & hot properties, surface properties, anti-oxidant properties and thermal stability, anti-wear and EP properties, and anti-corrosion properties.

3.2 Performance tests

3.2.1 Cutting fluids performance test results – turning tests

The results concerning tool flank wear and workpiece roughness are illustrated in Figure 4; the tests have been performed under two different conditions with a the depth of cut of 0.3 mm (on the left) and depth cut of 0.5 mm (on the right). At least two repetitions have been performed with each oil, the reference cutting oil SUPRACO 4018 and the new developed oil FIEC 13024. In Figure 4, the approximate medium values in both cases are showed.

Figure 5 collects the pictures of an example of flank wear after every two cuts with the different inserts tested under both machining tests and with both comparative oils.

From these tests it can be concluded that the Turning tests under 2 different cutting conditions with the two studied cutting oils have offered similar results concerning tool wear, which is the main aspect to take into account regarding the general machining performance. Regarding the workpiece roughness, also similar results have been obtained in the case of depth: 0.3 mm and some small variations in the case of depth: 0.5 mm, where the piece is supporting more aggressive cutting conditions; but this difference could be assumed within the scatter range generally associated with this kind of machining tests.

3.2.2 Hydraulic fluid performance test results

Ball on disc abrasion tests (rotational motion)

The ball-on disc abrasion tests results are summarized in Figure 6: the graphic on the left presents the friction coefficient of the tested fluids versus time and in the table of the right the average wear of the disc and ball are represented. The aspect of the wear scar obtained with each fluid can be seen in Figure 7.

It can be concluded that under the selected testing conditions the new developed hydraulic fluid BESLUX HIDRO-ECO-46 has better behavior than the reference mineral based hydraulic fluid BESLUX HIDRO-ECO-46.

FZG Test results

- Failure load stage: >12.
- Pinion torque at failure load stage: >534.5 N/m.

It can be concluded that the BELSUX HIDRO-ECO 46 oil has reached the maximum load stage defined in DIN ISO 14635-1 without scuffing failure appearance.

3.2.3 Grease performance test results

In Figures 8 and 9 summarized the test results obtained.

In Figure 8 it can be seen that the friction coefficient obtained with the VOSOLUB grease is much lower that the COF obtained with the reference mineral grease. It must be underlined that under test 2 conditions (2000 N) the tests has been stopped before finishing, the high presence of pitting has produced great vibrations on the system.

In Figure 9 the wear suffered by the rail and the wheel can be observed. During the first test the wheel and the rail suffer an initial wear mechanism of surface polishing. But when increasing the load, a fatigue phenomena appears, which translates into the pitting and micro-pitting suffered by the samples. The wear track obtained in the wheel when testing with

Table 1. Analytical results on main specification requirement on based VHOSO oil.

Properties	Tests	Unit	Methods	Results	Specification requirements
Composition and characteristics	Fatty acids composition and content	% (m/m)	ISO 5508/5509	87.5	Min 87% oleic acid
	Tocopherols content	mg/kg	ISO 9936	691	To be reported
	Triglycerides polymers composition and content	% (m/m)	ISO 16931	ITERG	Max 3
	Iodine value	cg I_2/g	ISO 3961	85	Max 100
	Peroxide value	méqO_2/kg	ISO 3960	0.9	Max 1.5
	Metals by ICP-OES	mg/kg	ASTM D 5185	All < 1	Excepted Exepted *P*, other metals < 3
	Phosphorous content by ICP-OES	mg/kg	ASTM D 5186	<1	Max 50
	Acid number	mg KOH/g	ASTM D 664	0.05	To be reported
	Water content	% (m/m)	Karl Fisher	0.009	Max 0.1
Viscosity	Kinematic viscosity at 0 °C	mm^2/s	ISO 3104	Gelified	Convenient for formulated products
	Kinematic viscosity at 40 °C	mm^2/s	ISO 3104	39.86	
	Kinematic viscosity at 100 °C	mm°/s	ISO 3104	8.546	
	Viscosity index		ASTM D 2270	200	Min 190
Cold properties	Cloud point	°C	ASTM D 2500	−13	To be reported X
	Pour point	°C	ISO 3016	−15	Min −15 (Better −20 °C)
	Low temperature stability 7 days at −10 °C	Viscosity change (%)	ASTM D 2532	Gelified	Initial viscosity ± 10%
Hot properties	PMCC flash point	°C	ASTM D 93	>300	Min 200
	COC flash point	°C	ASTM D 92	322	Min 220
	Fire point	°C	ASTM D 92	352	Min 250
	Autoignition point	°C	ASTM E 659	423	Min 350
	Noack volatility loss at 150 °C	% (m/m)	ASTM D 5800 mo	0.1	Max 2
Surface properties	Foaming characteristics	ml/s	ISO 6247	10-0/10-0/10-0	50-0/20-0/50-0
	Air release value	Min	ISO 9120	<1	Max 7
	Demulsibility	ml oil/ml wat/ml emu (min)	ISO 6614	41/39/0 (15)	40/40/0 (30)
Anti-oxidant properties and thermal stability	Baader ageing at 95 °C – 72 h (viscosity change)	%	DIN 51554-3	23.18	Max 10
	Rotating Pressure Vessel Oxidation Test (RPVOT) at 120 °C without water	Min	ASTM D 2271	47–46	Min 49
	Oxidation rancimat at 110 °C	Hours	ISO 6886	27.7	Min 25
Anti-wear and EP properties	Four-balls wear test	mm	ASTM D 4172	0.62	Max 0.5
	Welding load by four-balls test	kg	ASTM D 2783 mo	126	Min 126
Anti-corrosion properties	Copper corrosion test	Cotation	ISO 2160	1a	To be reported
	Steel corrosion test	Cotation	ISO 7120 A 4h	Fail (20% surface)	To be reported
	Steel corrosion test	Cotation	ISO 7120 A 24h	Fail (30% surface)	To be reported
Compatibility with elastomers	Hardness change	Shore A	ISO 6072 NBR1-HNBR-FPM AC6-AU	0.8/−0.1/1.0/0.5	±10
	Volume change	%		4.8/2.4/1.0/−1,2	− 3 to + 10
	Change in elongation	%		−81/−38/−2/63	Max 30
	Change in tensile strenght	%		−71/4/2/61	Max 30
	Filtrability	FI	NF E 48-690	Plugged filter	Max 1.5
Environmental behaviour	Ultimate biodegradability	%	OECD 301B	83.7	Min 60
	Acute toxicity on fishes, LC 50	WAF mg/l	OECD 203	>1010	Min 1000 mg/l WAF
	Acute toxicity on daphnia, IC 50	WAF mg/l	OECD 202	>1010	Min 1000 mg/l WAF
	Acute toxicity on algae (growth inhibition), EC 50	WAF mg/l	OECD 201	>1050	Min 1000 mg/l WAF

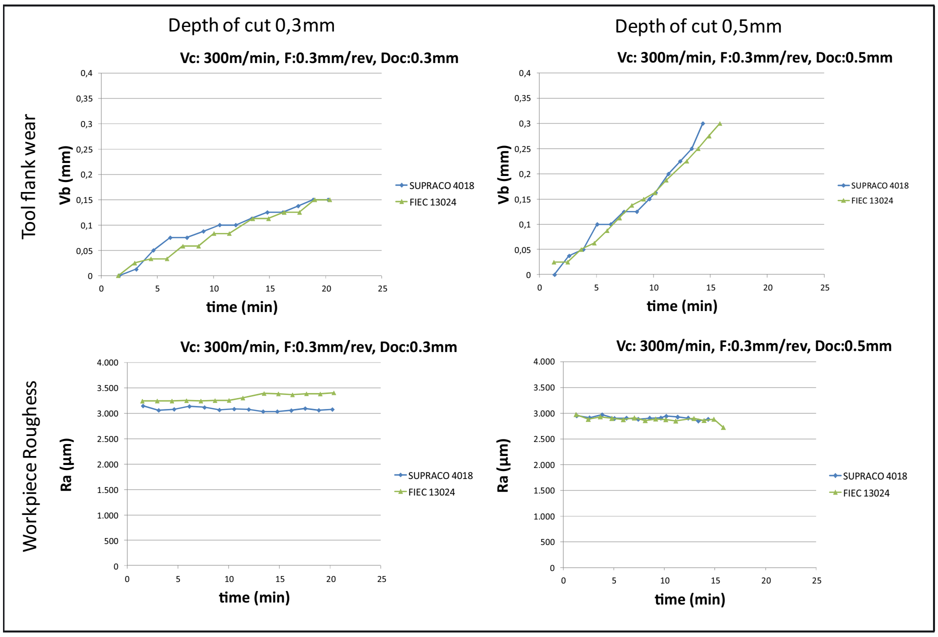

Fig. 4. Workpiece roughness (up) and tool Flank wear (down) mean values when using a cutting depth of 0.3 mm (left) and a cutting depth of 0.5 mm (right).

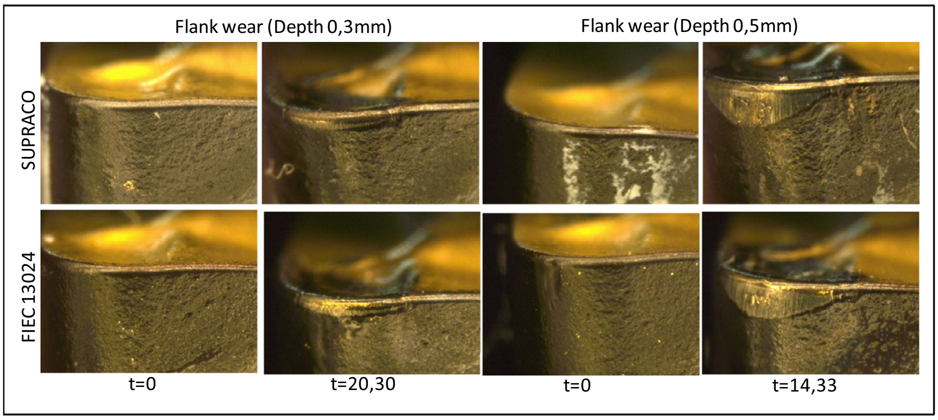

Fig. 5. Flank wear obtained with the reference cutting fluid (up) and new developed fluid (down), when using a cutting depth of 0.3 mm (left) and cutting depth of 0.5 mm (right).

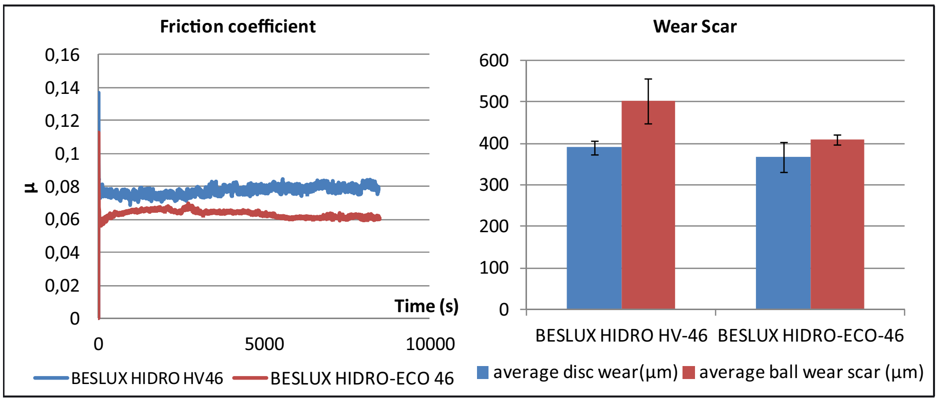

Fig. 6. Friction coefficient and wear scar of the reference mineral fluid (HV-46) and the developed fluid.

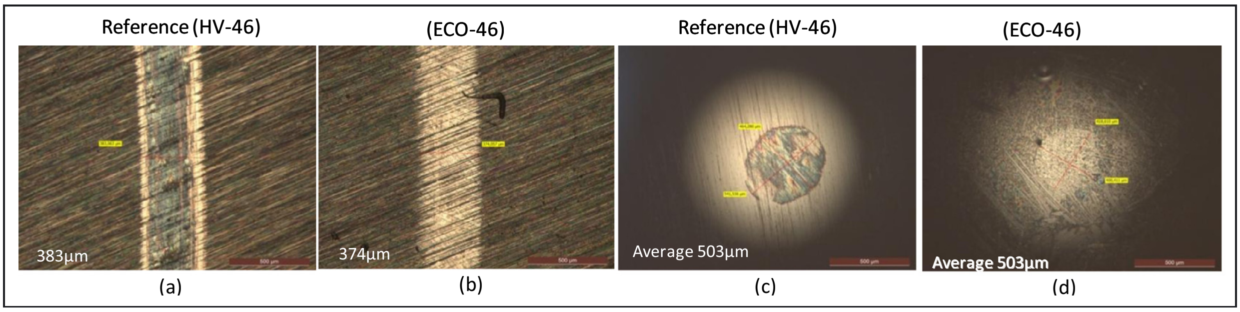

Fig. 7. Ball (c, d) and disc (a, b) wear scar obtained with reference fluid (a, c) and the developed ECO fluid (b, d).

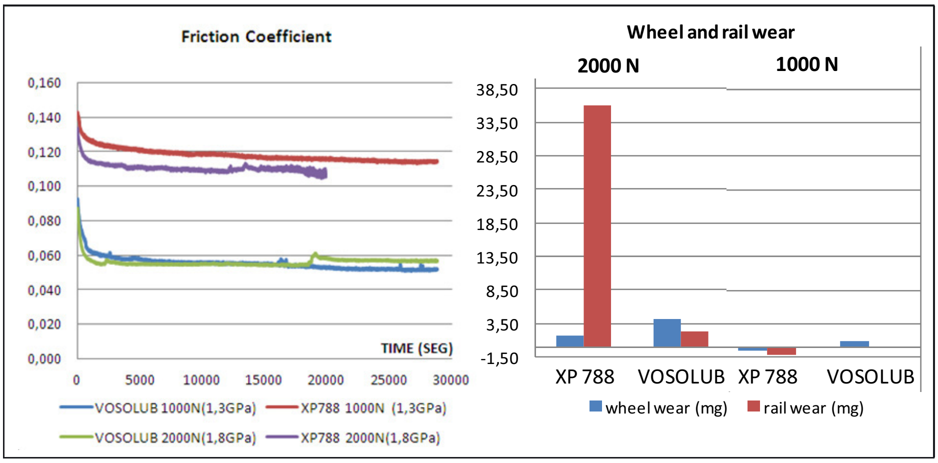

Fig. 8. Friction coefficient of greases (left) and wheel and rail wear (right).

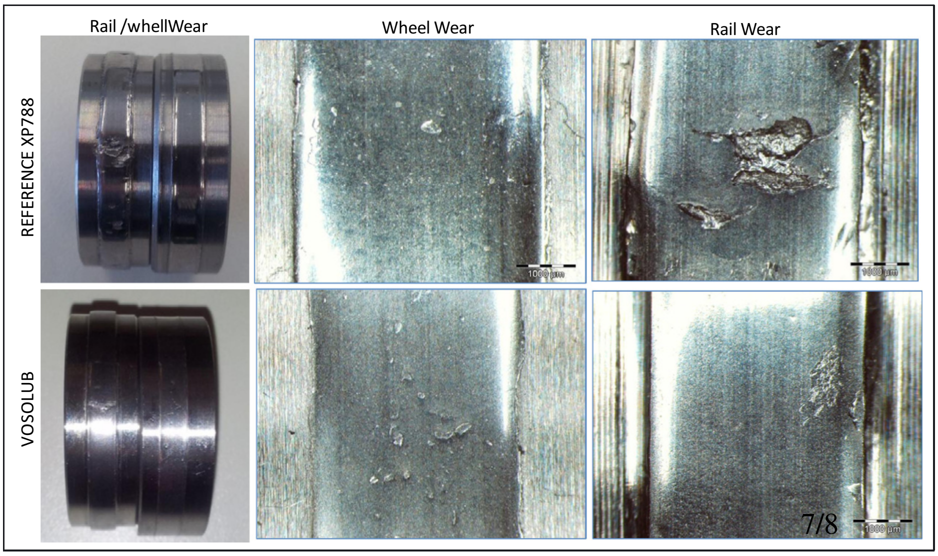

Fig. 9. Wheel and rail wear.

the reference XP 788 grease is deeper than the wear track obtained with the developed VOSOLUB grease. In both wheels also some micropitting is observed.

Concerning the rail, when testing with the VOSOLUB grease, it suffers some micripitting throughout all the rail. In addition this rail also presents 1 pitting failure. On the other hand, the rail tested with the reference XP788 grease suffers important pitting throughout all the rail.

– The friction coefficient of the new developed VOSOLUB grease is much lower than the friction coefficient obtained with the reference grease. Not great differences have been observed in the friction coefficient in the different testing conditions.
– Concerning the wear, the XP788 grease suffers higher wear than the new developed grease in terms of mass loss, and also suffer much more pitting and micropitting.

- It can be concluded that under the selected testing conditions new developed VOSOLUB grease has better behavior than the reference mineral XP788 grease.

4 Conclusion

Based refining VHOSO oil used for biolubricant formulation match specification required. Results on performance tests done on the three targeted applications comply with requirement expected by the formulators. On this base, "field tests" on real condition have been carried out by formulator to assess technical and marketing aspect of "VOSOLUB biolubricant". On the same time developments to obtain Ecolabel are in progress. First results of "field tests" currently in progress are very encouraging and conditions for a successful market uptake of innovative biolubricant are there.

- MOTUL TECH (France): Biobased products with high and long-term performances and a safe HS profile for cutting oil Application.
- BRUGAROLAS (Spain): Biobased hydraulic fluid, potentially ecolabelisated, with high performing properties, ecologically safe in case of incidental release to the environment.
- Rs CLARE (UK): Ecologically safe, biodegradable lubricant for a Curve side-of-rail lubricant application that has total loss to the environment, while maintaining existing performance levels.

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