WHITE PAPER

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BIO-GREASES:

What everyone wants but nobody buys

The EU Eco-label

Heavy Duty Lubricant from Sunflowers

AXEL

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BIO-GREASES: what everyone wants but nobody buys!

In considering the environmental effect of different products there is often confusion about what is actually relevant and, over the years, a whole array of terms and concepts have developed which only serve to further complicate the situation. In principle, there are no chemicals, apart perhaps from pure water, which are generally good for the natural environment (and even with water, this can depend on the amount in question). It is therefore a mistake to refer to environmentally "friendly" products. This has clearly been recognised and new categories of products are being promoted as being environmentally suitable, environmentally acceptable, environmentally adapted, environmentally compliant and most recently, environmentally responsible.

When it comes to lubricants, the whole concept of environmental compliance has become a general term meaning *types of fluids which rapidly decompose in water and soil*. And the question then becomes "compliant to what"? It is obviously not enough to only measure some degree of biological degradability to be able to decide whether or not a lubricant is environmentally acceptable. Other factors such as eco-toxicity in different media and bioaccumulation have also to be considered and vast complexities of rules and regulations have been imposed in different geographical regions. Examples of such "positive" classification systems can be the Nordic Swan, the German Blue Angel and the American Green Seal, all with different judgement criteria.

Lubricants, and especially lubricating greases, are very complicated in nature and finding relevant test methods and limiting criteria can be a very difficult job. The most ideal situation would be to have extensive data on every single finished product but normally it has to suffice with information on the individual components used in the formulation. A fully formulated grease is often insoluble in the chemicals (solvents) defined in the official test methods and this makes it often extremely doubtful whether the results measured on the finished product can be regarded as relevant and/or reliable. Making judgements on the component level can also be misrepresentative since some of them do not exist in the finished grease; they are reacted together *in situ* to produce the thickener system (mostly metal soaps) which provides the

HEAVY DUTY LUBRICANT FROM SUNFLOWERS

In recognition of the importance of the use of biobased products in industrial applications, a project, partly financed by the European Union, was set up to investigate the feasibility of using a natural vegetable oil as the base fluid in a heavy duty lubricating grease. During the late 1990's, refinements in plant biology made it possible to increase the amount of oleic acid in sunflowers. From these flowers, in this particular case a product of Spain, a vegetable oil commonly known as HOSO (High Oleic Sunflower Oil) may be extracted. This high content of oleic acid allows the commercial use of the oil in a wider temperature range compared to previous vegetable oils, being more resistant to oxidation and having a lower pour point. Compared to standard lubricating oils such as mineral oils or synthetic PAOs, the main advantages are biodegradability and renewability.

The project has focussed on replacing two different greases commonly used in the total loss physical matrix of the product. So either way, there are doubts and limitations.

In determining the environmental impact of a lubricant, it is important to be aware that there are two very different issues to be considered and these can be referred to as the primary and secondary effects. The primary effect is, of course, the direct and tangible results of the materials coming into contact with the natural environment, water pollution, for example. The secondary effect is achieved through the *use* of the product in different applications, lower energy consumption, longer life of mechanical components etc. These secondary effects can be the result of improved tribological systems and optimised lubrication without taking into consideration the bio-criteria normally associated with environmentally acceptable products. It is for the primary effects we need so-called bio-greases and especially in applications where these cannot be prevented from leaking out into the natural environment. This is often referred to as *loss lubrication*.

BIO-GREASES ARE INTENDED TO MINIMISE THE DETRIMENTAL EFFECTS OF INCIDENTAL LEAKAGE INTO SENSITIVE NATURAL ENVIRONMENTS

Already in 1992, the National Chemicals Inspectorate of Sweden published a comprehensive report on the chemical characterisation, functional features and health and environmental risks of lubricants. Authored by Jan Ahlbom and Ulf Duus, “Ren Smörja i Göteborg” (Clean Lubricants in Gothenburg) has become an international point of reference (ExxonMobil have, for instance, named the “Gothenburg Group” as one of the six most environmentally promotional projects in the world as regards lubricants) and the “Ren Smörja” requirements have been accepted as a Swedish Standard for hydraulic oils and greases. One conclusion of the report was that large amounts of

lubrication on large excavators. Apart from offering the same level of performance, the product developed was to be environmentally acceptable according to Swedish standard SS 15 54 70, class B (”Ren Smörja”). In addition, the benefit of having one grease instead of two was something that clearly interested the end user. The general objective of the project from an EU point of view was to increase the utilisation of natural ”re-growing” oils in non-food applications. Benefits included the increased use of agricultural products in industrial applications, increasing the agricultural land utilisation and creating jobs.

The environmental improvements and the decrease in the need for mineral oil lubricants derived from petroleum, were additional advantages.

To be able to fulfil this task, almost every aspect of the grease(s) needed scrutiny. The oil blend, the additive package, the thickener system and finally getting all the components to work in a synergistic fashion provided an interesting challenge. The final test was to be in a full scale field trial in a medium sized excavator working under harsh conditions in a quarry.

lubricants are inevitably lost into the environment each year and there is an urgent need to re-design these loss lubricants to be as harmless as possible for man and the environment. In 1997, the health/environmental criteria for lubricating greases were presented. These were primarily addressed to certain lubricating grease applications where the loss is significant and the product ends up in the natural environment. Examples of this can be centralised lubrication systems on heavy vehicles, lubrication on heavy off-road equipment, rail phlange lubrication and open gear lubrication. With "Ren Smörja" came a new demand over and above the general bio criteria, the use of renewable primary produce instead of the depletion of our natural finite resources such as mineral oil. So environmentally responsible lubricants should not only be biodegradable, non-toxic and non-accumulating but also based on renewable products such as vegetable oils and their derivatives.

In the absence of a global harmonisation system for the classification of lubricants formulated with regard to a minimal impact on the natural environment, different systems have been adapted in different parts of the world. In Europe, there are still many local and national programmes but more and more attention is being paid to the European Union Eco-label where lubricants have now been introduced as a new product group. In simplistic terms, to qualify for the European Eco-label, a product should be in compliance with six specific criteria: No "R-phrases", aquatic toxicity requirements, biodegradability and bioaccumulative potential, the exclusion of specific substances, renewable raw materials and, last but not least, technical performance. This will be dealt with in more detail elsewhere in this publication. The testing required for verification of compliance is comprehensive and, perhaps more importantly, very expensive indeed and this will most probably act as a barrier to a more widespread implementation of the system. In the United States, there has been a somewhat more pragmatic approach where the promotion of the use of biobased products gained a significant boost when the Farm Security and Rural Investment Act of 2002 (FSRIA) was signed into law. This has led, for instance, to the Federal Biobased Products Preferred Procurement Programme (FB4P) where federal agencies are required to purchase biobased products if possible. With reference to the definition of a biobased product, one phrase used has been "renewable domestic agricultural materials". In an attempt to clarify this concept, the UDSA has ruled that it should be based on the amount of biobased carbon as a percentage of the total organic carbon in the product. The clarification of the term "domestic" has also become a very sensitive issue. The USDA will also, where appropriate, recommend the minimum level of biobased material to be contained in the procured product and requires that agencies

Partners in the project were:

Fundacion Tekniker (Spain): administration, tribological & general testing, biodegradability and eco-toxicology.

Liebherr (France): application knowledge, end user, field testing.

RheinChemie (Germany): additive development, general rig testing.

Hydromechanique et Frottement R&D (France): application knowledge, field test simulation

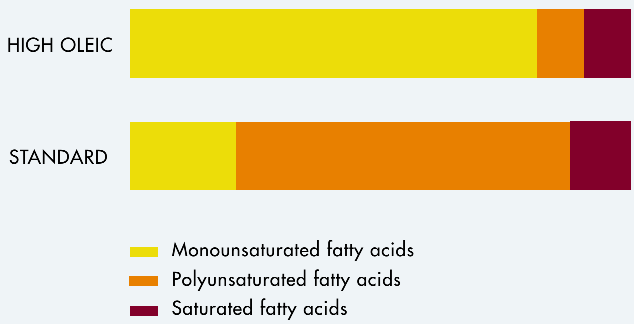
Axel Christiernsson AB (Sweden): base fluid and grease development, general testing.

More specific goals were to improve the performance of the vegetable oil to make it suitable as the base fluid in an environmentally acceptable lubricating grease, to utilise a new and advanced thickener system, to implement the latest additive technology, to increase the general performance of the grease to achieve an extension of the component lifetime, to perform a full life cycle analysis of the complete product purchasing designated items give preference to those products that have the highest percentage of biobased products practicable. Other acceptable criteria for environmental acceptability include life cycle cost analysis verified by a "BEES" analysis or ASTM D 7075 standard for evaluating and reporting on environmental performance of biobased products, including life cycle costs. ("BEES" is an acronym for "Building for Environmental and Economic Sustainability", an analytical tool used to determine the environmental and health benefits and life cycle costs of items).

So, different approaches in different places and this can cause havoc for international companies operating on a global basis where product stewardship requires identical formulations for specific brands no matter where in the world they are manufactured or sold.

So progression from a fundamental and understandable drive to implement the use of bio-greases into any real market breakthrough is slow, so very slow. Biogreases, what everyone wants but nobody buys.

HIGH OLEIC SUNFLOWER OIL COMPARED TO ORDINARY SUNFLOWER OIL



chain, to fulfil the specifications for greases used in excavators in a new multifunctional biobased product, to fulfil the criteria in SS 15 54 70, class B and, if possible to develop a product suitable for use in other types of applications.

In implementing the use of HOSO as the base fluid, several problems needed to be overcome. Pure HOSO solidifies rapidly at -12°C with the formation of a dense wax network. A better test method for low temperature stability was needed since pour point was not deemed to be a relevant method. The viscosity of HOSO was far too low compared to the specified level and needed to be boosted but the oil is unfortunately incompatible with many of the polymers available. In addition, the use of a viscosity modifier tends to deteriorate the low temperature performance. The good ecological and toxicological properties of HOSO were to be maintained. Eventually, by combining specific polymers with a special di-ester, a viscosity modifier was introduced and the final base oil developed had the desired properties: Viscosity/40°C = 200 mm^2/s, VI > 180, Fluid for 20 days @ -22°C, Maintained biodegradability (HOSO content > 80%).

The EU-Eco-label

The European Union Eco-label, a unique certification system established in 1992, is a voluntary scheme designed to encourage businesses to market products and services that are preferable from an environmental perspective. The "flower" is the symbol of the European Eco-label, a guide for consumers, including public and private purchasers, to identify "greener" alternatives.

Over the past decade, the flower has become well known as a pan-European product symbol which gives the consumer simple and exact guidance. All products carrying the Eco-label, symbolised by the flower, have been reviewed by independent bodies to guarantee that they fulfil the stringent environmental and functionality criteria. The European Eco-label is part of a broader strategy aimed at promoting sustainable consumption and production. The flower can be found not only throughout the whole European Union but also in Norway, Iceland and Liechtenstein. At present, there are 23 different product groups and more than 250 licences have been granted for several hundred products.

Lubricants is a relatively new product group and the work has been led by the Dutch competent body. Starting with a first Ad-hoc working group formed on December 11, 2003 in Brussels, the European Commission has now come to a decision establishing ecological criteria and the related assessment and verification requirements for the award of the Community eco-label to lubricants (document number C(2005) 1372, April 26, 2005). Since the use of lubricants may be hazardous for the environment due to, for example, their aquatic toxicity or their bioaccumulation, it was deemed that appropriate ecological criteria should be laid down. The product group "lubricants" shall comprise hydraulic oils, greases, chainsaw oils, two stroke engines, concrete release agents and other total loss lubricants, for use by consumers and professional users. In order to be awarded the Community eco-label for lubricants, it must

The thickener technology chosen was a new and innovative polymer thickener providing great opportunities to design a properly functioning grease. The non-ionic character of this newly patented thickener provides a superior additive response compared to soap thickened greases. The thickener is inert and is compatible with the more traditional types of grease. Different additive systems were composed and tested before the final formulation was established.

Despite formulating the first prototype to meet the originally proposed specification (basically drawn up from the two products currently in use), the grease did not work very well in the simulation tests, and there was a clear need for further optimisation. In addition to a small recipe adjustment in the grease itself, (providing a slight increase in oil separation), the conditions of the tribological tests had to be adapted to better match the real working conditions. Once this was done, a clear improvement could be seen.
In a final round of tribological testing and field trial simulation, the second prototype was shown to be as good as, if not better than the products currently used. In the two different types of machine elements present on modern excavators, both gears and pin & bushings, the new grease

comply with the criteria set out in the annex to the decision. The criteria shall apply to the freshly manufactured product at the time of delivery. These criteria aim in particular at promoting products that pose the least harm to the water and soil during use and which lead to reduced CO_2 emissions.

In simplistic terms, to qualify for the European Eco-label, a product should be in compliance with the following criteria:

No "R-phrases": The product shall not have been assigned any R-phrase at the time of applying for the eco-label, indicating environmental and human health hazards according to the 1999 Dangerous Preparations Directive.

Aquatic toxicity requirements: Data is required on the aquatic toxicity of both the preparation and its main components and limits have been set for different product subgroups. For greases, for instance, the critical concentration for the aquatic toxicity shall be at least 1000 mg/l.

Biodegradability and bioaccumulative potential: The product shall not contain substances that are both non-biodegradable and (potentially) bioaccumulative. However, the product may contain one or more substances with a certain degree of degradability and potential or actual bioaccumulation up to an indicated cumulative mass concentration.

Exclusion of specific substances: Substances appearing in the Community list of priority substances in the field of water policy and the OSPAR List of Chemicals for Priority Action shall not be intentionally added as an ingredient in a product eligible for the Community eco-label. Other excluded products are organic halogen and nitrite compounds and even metals or metallic compounds (with the exception of sodium, potassium, magnesium and calcium). In the case of thickeners in greases, lithium and/or aluminium compounds may be used up to concentrations limited by other criteria in the annex.

Renewable raw materials: The formulated product shall have a carbon content derived from renewable raw materials exceeding a certain percentual limit for different product sub-groups. For greases, this should be more than 45% mass.

Technical performance: The products should meet given prestanda requirements which are specific to the different sub-groups. For greases, they should be "fit for purpose", whatever that may imply!

performed really well. In neither case was the use of solid lubricants seen to be beneficial. In the bio-tests, the final formulation was seen to be environmentally acceptable. Primary biodegradability (OECD 301) being in excess of 88% and all the components of the grease presenting good inherent biodegradability (OECD 302) as long as the total amount of polymer and additives did not exceed 10%. In the eco-toxicity test (OECD 202 *Daphnia Magna* acute immobilisation test), all the components showed values classed as non-harmful.

In this project, it has been demonstrated that a new "bio-grease" based on renewable HOSO can replace two completely different types of lubricating grease used on a modern excavator. BIOGREASE was an EU framework project aimed at generating industrial uses for biological material, partly funded by the EC under the contract number QLK5-2000-00611.

More information on the project and the product can be obtained by contacting the AXEL project manager, Mikael Kruse: mikael.kruse@axelch.com

In the next issue of the White Papers, we will address the issues of heavy load applications and how the grease manufacturers' response has progressed over the years through the incorporation of advanced additive systems and the development of functional soaps.

As usual we encourage our readers to give us feed-back and requests for grease technology topics they want us to cover in future Lubrisense White Papers..
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