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Tribological Performance of Ionized Vegetable Oils as Lubricity and Fatty Oiliness Additives in Lubricants and Fuels

Lubricity and oiliness additives, also known as friction modifiers, are steadily gaining acceptance from lubrication engineers and lubricant formulators. This paper describes how describes how such additives function in various tribosystems and which parameters control lubricity of finished formulations. Extensive experimental data are presented to demonstrate the outstanding tribological performance of bio-based lubricity and fatty oiliness additives produced by Elektrionization™ of vegetable feedstocks. Featuring a unique combination of viscosity and polarity, ionized vegetable oils form sufficiently thick and resilient protective layers by adsorption to rubbing surfaces. It is shown that, unlike extreme pressure additives, which act when a direct asperity-asperity contact occurs in the boundary lubrication regime, ionized vegetable oils function by postponing the onset of the boundary lubrication regime.

Introduction

The terms "lubricity" and "oiliness" refer to slipperiness of lubricant films formed in boundary lubrication, a condition which lies between unlubricated sliding and fluid-film lubrication, and which is also defined as that condition of lubrication in which the friction between the surface is determined by the properties of the surfaces and properties of the lubricant other than viscosity. Boundary lubrication encompasses a significant portion of lubrication phenomena and commonly occurs during the starting and stopping of machines.

The function of lubricants in tribological systems is to reduce friction and wear. The reduction of friction results from the formation of a lubricant film separating the rubbing surfaces. As a rule of thumb, lubricant films are anisotropic and reveal fairly complex non-Newtonian rheology. As a result, the coefficient of friction is not constant - it depends on the applied load, film deformation (elastic effects), sliding speed (viscous effects), as well as on their time-derivatives.

The thickness of the lubricant film depends upon constituent chemistry (base oil and additives), as well as upon the operating conditions, specifically the applied load and the sliding velocity. At a sufficiently high load or low sliding speed - the condition encountered e.g. for rod journals near the top dead center - lubricant may be expelled from the friction zone, leaving the rubbing surface unlubricated. In this case, severe friction and intense wear result.

Previously, in the field of fuels and lubricants, lubricity had always been taken for granted. However, the situation has started to change in the past two decades. Lubricity - or rather the lack thereof - has become a "hot topic" in the beginning of the 90s, following the introduction of ultra-low sulfur diesel (ULSD).[1,2] The major benefit of the change to ULSD is that the environmental impact of emissions of sulfur dioxide is greatly reduced. However, soon it has been realized that a reduction in the sulfur content also causes a reduction in the fuel lubricity, blamed for premature fuel pump and injector wear. To address this new challenge, ASTM has developed the lubricity standard ASTM D975 which went into effect on January 1, 2005.

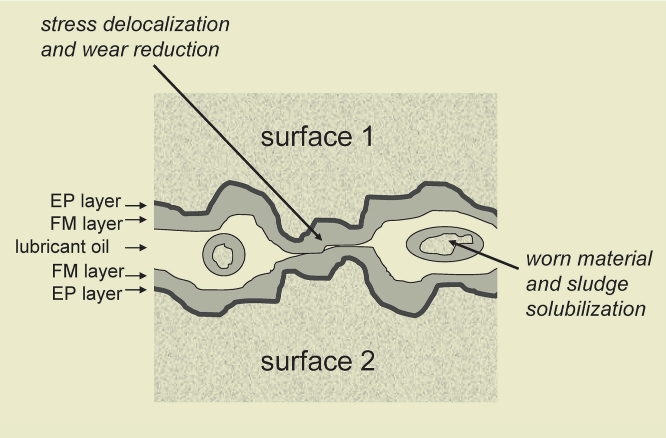
At the same time, in the lubricant branch, ever-growing quality demands and stringent environmental regulations have led to broad commercialiazation of hydrocracking, catalytic dewaxing and hydrofinishing technologies creating an abundant supply of API Group II and III base oils. The soaring crude oil price also drives development of other competing technologies, such as gas-to-liquid (GTL) conversion using the Fischer-Tropsch process. However, despite many undisputed advantages over their Group I predecessors, new base oils produced using the all-hydrogen route or the GTL conversion have one major drawback - they lack solvency and lubricity.

To alleviate the dramatic effect of "dry" friction, extreme pressure (EP) additives are deployed. Those additives - normally containing sulfur, phosphorus or chlorine - are capable of reacting with the material of rubbing surfaces to form a thin surface layer of sulfide, phosphate or chloride, which acts as a solid lubricant when the rubbing surfaces come into a direct contact with each other. When unlubricated sliding is encountered, friction and material deformation generate enough heat to trigger the EP reactions. However, the price of that is a microscopic scar left at the surface. Such scars are accumulated with time to give wear.

It is important to realize that EP additives start to act after the asperity-asperity contact has occurred - but they do not prevent its occurrence. The latter task is left to friction modifiers - compounds which form soft but sufficiently resilient protective layers by adsorption to rubbing surfaces.[3] Friction modifiers, also known as lubricity and oiliness additives, are steadily gaining acceptance from lubrication engineers and lubricant formulators. Depending on the molecular structure and chemistry of friction modifier, a brush-like dense monolayer or a sponge-like diffuse multilayer will form; the latter is capable of retaining base lubricant in the friction zone. When the protective layer is mechanically deformed under the applied load, a disjoining pressure builds up pushing the rubbing surfaces apart. A number of theories exist attempting to explain the mechanism of short-range sterical repulsion between surfaces containing adsorbed layers of polar components.[4]

Most readers of this paper have probably experienced such a lubricity-enhancing effect while walking on the slippery rocks of the seashore. The algae slime growing on the rocks retains a sufficiently thick layer of water which acts as a lubricant between your feet and the rock surface. The mechanism of action of such a friction modifier is explained in Figure 1.

Figure 1



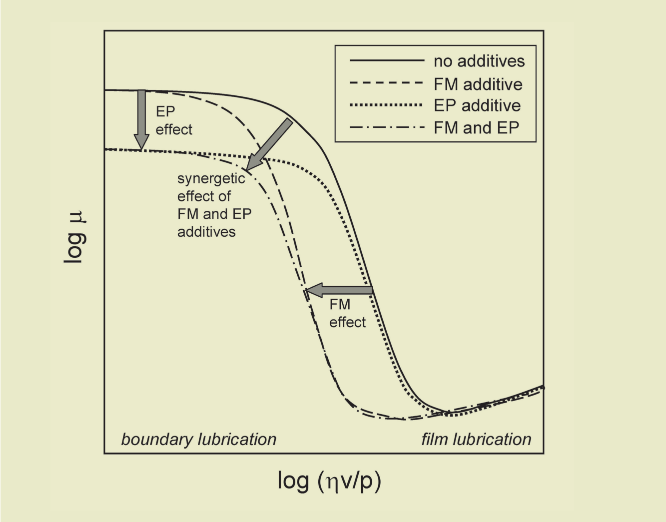
The formation of such a surface film has a beneficial tribological effect as it reduces friction and wear, as well as associated energy dissipation, noise generation, and tribomutation. It is interesting to note that a lubricity-enhancing effect may also be achieved by surface texturing, whereby a system of surface pores is generated to keep lubricant in friction points.[5]

A good friction modifier is expected to combine a sufficiently high adsorptivity to metal surfaces (which is largely controlled by its polarity) and the ability to form a sufficiently thick and resilient protective film (which is basically determined by its viscosity), as well as the ability to dissolve in base oils where it is meant to be used in. These are antagonistic properties, as increasing polarity reduces miscibility with mineral base oils. For instance, polyethylene glycols are highly polar but immiscible with mineral oils. Heavy polyalphaolefins, on the contrary, are non-polar at all - as a result, they have no surface activity whatsoever. Many synthetic esters also lack polarity and may desorb with increasing oil temperature, leaving the surface unprotected. Thickness of the adsorbed layer normally increases with increasing the viscosity, which is linked to increased average molecular weight and intermolecular cohesion. Molecular structure has significance, too - most friction modifiers have amphiphilic molecules similar to those of surfactants. Other important factors are thermal stability, oxidation stability, as well as ecological and health safety considerations.

The majority of base oils are the so-called normal fluids demonstrating nearly Newtonian rheological behaviour at low sliding velocities. In this case, even moderate loads may displace the oil from the friction contact zone. For instance, in a car engine, the cylinder liner-piston ring tribosystem has two dead points in which load are combined with zero sliding velocity. At those points, thinner fuel-economy engine oils - such as those of 0W-20 specification - may fail to provide adequate film thickness unless appropriate film-thickening lubricity components are included in their formulations.[6-9]

Unlike extreme pressure additives, which act when a direct surface-to-surface contact occurs in the boundary lubrication regime, friction modifiers function by postponing the onset of the boundary lubrication regime as explained in Figure 2.

Figure 2



Equivalently, one may talk about expanding the borders of the hydrodynamic lubrication regime: in a loading cycle (when moving from the right to the left over the Stribeck curve), the film lubrication will stay longer and stand higher loads, and in an unloading cycle (moving from the left to the right), the change from boundary lubrication to hydrodynamic lubrication will occur earlier.

Friction modifiers can also be regarded as mild antiwear agents working at moderate temperatures and loads where conventional antiwear and EP additives, such as tricresylphosphate (TCP), zinc dialkyldithiophosphate (ZnDTP), and methylene-bis(di-n-butyldithiocarbamate) to mention a few, are not yet reactive.

The protective layers formed by friction modifiers are self-regenerating: if the layer is damaged by applying too high a stress, it will be restored by adsorption of a new portion of friction modifier from the bulk and by lateral diffusion of adsorbed molecules caused by a surface pressure gradient.

Goals of the present research

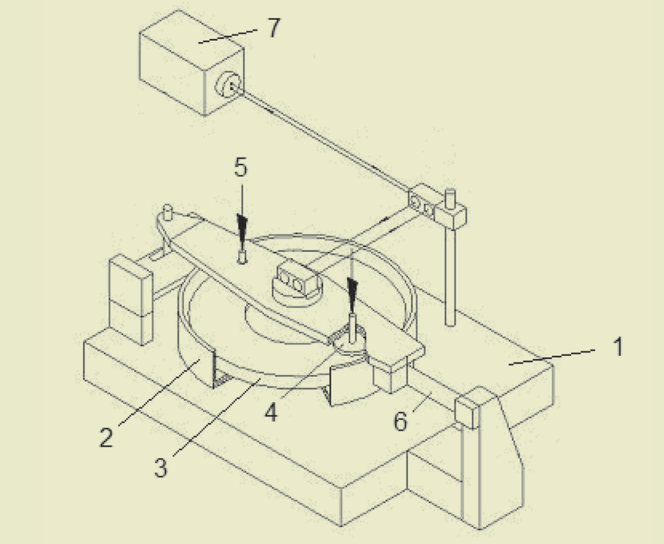
The primary goal of this paper is to demonstrate the outstanding tribological performance of bio-based lubricity and fatty oiliness additives produced by Elektrionisation™ of vegetable feedstocks, a proprietary process whereby feedstocks go through electro-ionizing treatment.[10-11] This leads to an increase in viscosity, polarity and viscosity index, due potentially to the branching of fatty chains and the formation of small amounts of free fatty acids and mono- and biglycerides. No catalyst is used, nor any synthetic additives. Increasing the polar functionality of molecules of vegetable oil has a positive impact on friction and wear protection resulting from stronger adsorption on metal surface as well as from the thermodynamic stabilization of the adsorbed layers by crosslinking and lateral interaction between adsorbed molecules.[12]

Experimental

Tribological tests were carried out using a Morel tribotester (steel vs steel) specially designed to enable accurate friction coefficient measurements in hydrodynamic, elastohydrodynamic and boundary lubrication regimes. The conceptual instrumental setup is outlined in Figure 3 and consists of two steel blocs pressed against a rotating steel disk immersed in the lubricants in study.

The rotation speed and the pressure applied to the plates can be varied, and the resulting torque produced by the friction force measured, whereby the coefficient of friction is derived as a function of the Hersey number (viscosity x sliding velocity / applied pressure). Testing results were presented in the form of Stribeck diagrams plotting friction coefficient versus Hersey number.[13]

Figure 2



The Hersey number may be considered as a measure of effective lubricant film thickness between the rubbing surfaces. The use of Stribeck plots allows a compact and consistent representation of data collected at various loads and sliding velocities. The actual pressure settings were 1.96, 3.92, 5.89, 7.85 and 9.81 bar (1 bar = 100 kPa). The test conditions were adjusted so as to cover the transition from the elasto-hydrodynamic (EHD) to the boundary lubrication regime.

Essential physicochemical characteristics of lubricants and fuels used in the tests are summarized in Tables 1 to 5.

TABLE 1) Characteristics of jet fuel (JP-8) used in tribological tests

Parameter	ASTM	Unit	Specification
Total acid	D3242	mg KOH/g	0.015
Aromatics	D1319	vol.%	25.0
Olefins	D1319	vol.%	5.0
Naphthalenes	D1840	vol.%	3.5
Sulfur	D1266	wt.%	0.3
Distillation	D86		
- point 10%		°C	205
- point 90%		°C	212
- point final		°C	300
- residue		vol.%	1.5
- loss		vol.%	1.5
Flash point	D93	°C	38
Pour point	D2386	°C	-47
Density at 15 °C	D1298	g/cm^3	0.8
Viscosity at -20 °C	D445	mm^2/s	8.0

TABLE 2) Viscosity of JP-8 fuel depending on the concentration of the additive and temperature

Temperature (°C)	Viscosity (cSt)		
	without E-ION R	0.1% E-ION R	0.25% E-ION R
30	1.682	1.704	1.794
50	1.194	1.214	1.256
80	0.838	0.852	0.868

TABLE 3) Characteristics of mineral oil (100N) used in tribological tests

Parameter	ASTM	Unit	Specification
Density at 15°C	D1298	g/cm^3	0.87
Viscosity at 40°C	D445	mm^2/s	22.8
Viscosity index	D2270	-	97
Flash point	D92	°C	210
Pour point	D97	°C	-12
Neutralization number	D974	mg KOH/g	0.03

TABLE 4) Viscosity of 100N oil depending on the concentration of the additive and temperature

Temperature (°C)	Viscosity (cSt)		
	without E-ION R	2.5% E-ION R	10% E-ION R
30	30.62	34.92	51.77
50	14.12	16.14	24.46
80	6.15	7.00	9.98

TABLE 5) Characteristics of E-ION R friction modifier

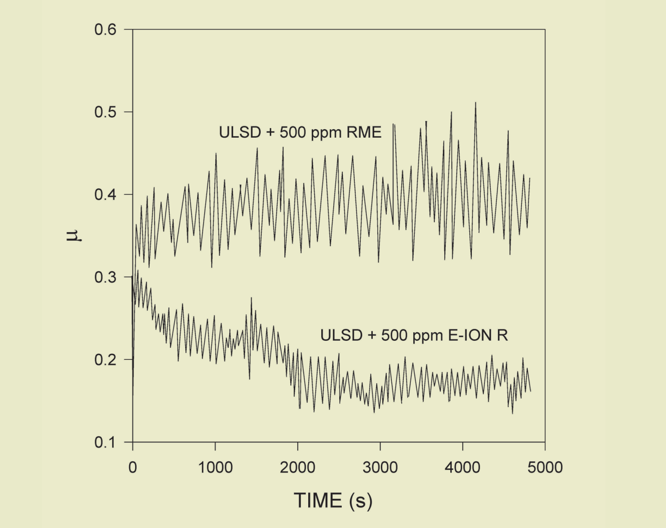
Property	ASTM	Unit	Typical value
density at 15°C	D4052	g/cm^3	0.909
viscosity at 40°C	D445	mm^2/s	2280
viscosity index	D2270	-	277
refractive index	D1747	-	1.482
pour point	D97	°C	-3
flash point	D93	°C	270
aniline point	D611	°C	85
sulfated ash	D874	wt.%	0.03
Conradson carbon residue	D189	wt.%	0.28
iodine number	D1959	$cg\ I_2/g$	38
total acid number	D664	mg KOH/g	1.4
saponification number	D94	mg KOH/g	89

Results and Discussion

(i) Fuel tests

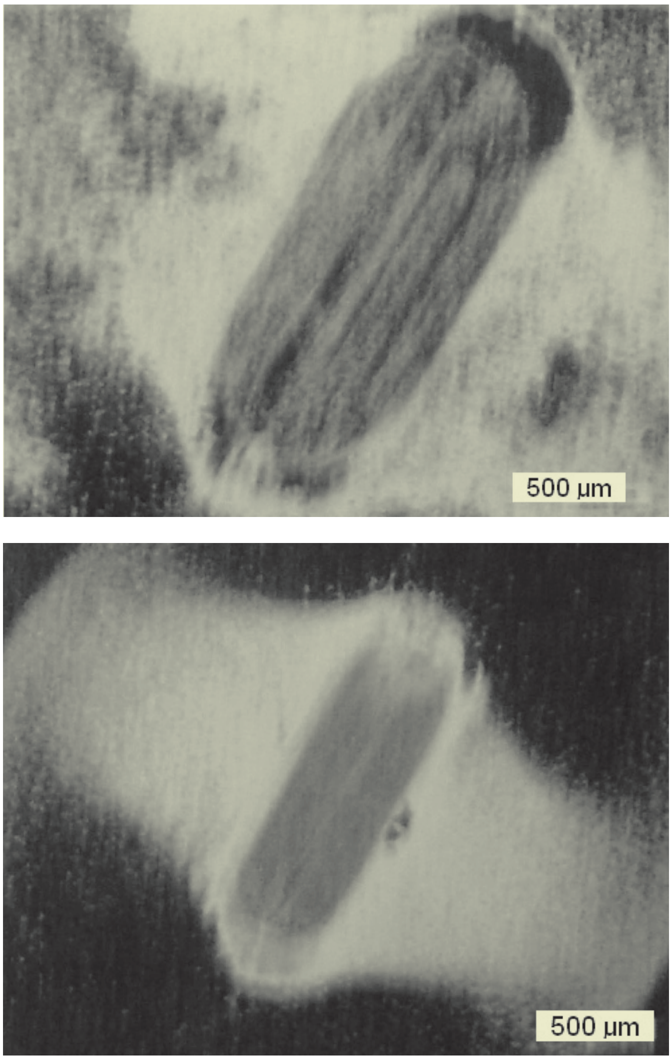
In the early 90's, lubricity has become a concern for fuels after the introduction of ultra-low sulfur diesel (ULSD).[1,2] By December 1, 2010, all highway diesel in USA will be ULSD. However, the first field experience with ULSD in Sweden was quite a disaster as numerous failures of fuel pumps and increased injector wear sulfur (<15 ppm in ULSD vs 500 ppm in LSD) also removed the were reported and later linked to the fact that the removal of beneficial EP effect of sulfur compounds. Nowadays, RME (rapeseed methyl ester, also called biodiesel) is most commonly used as a lubricity improver for ULSD (up to 5% addition levels).[14]

Figure 4



As can be seen in Figure 4 and 5, ionized vegetable oils outperform RME in terms of surface film thickness and antiwear efficiency.

Figure 5



The results in Figure 5, together with some independent studies and field trials, also indicate that ionized vegetable oils improve surface cleanliness thus reducing risk of injector fouling and filter blocking.

To study the effect of ionized vegetable oils on other types of fuel, tribological tests have been carried out for jet fuel (JP-8) doped with E-ION R. As expected, the addition of E-ION R shifts the beginning of the boundary lubrication regime towards lower Hersey numbers (see Figure 6).

Figure 6a

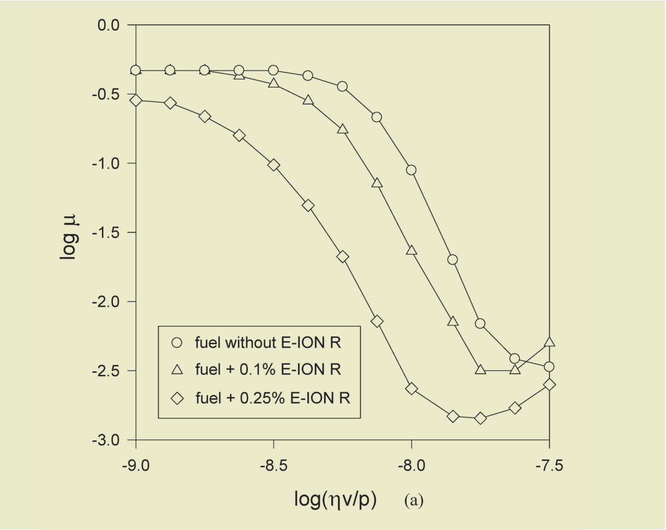
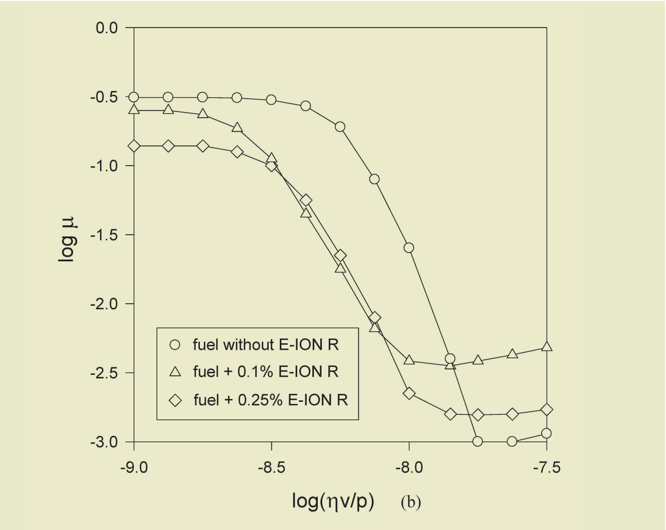


Figure 6a



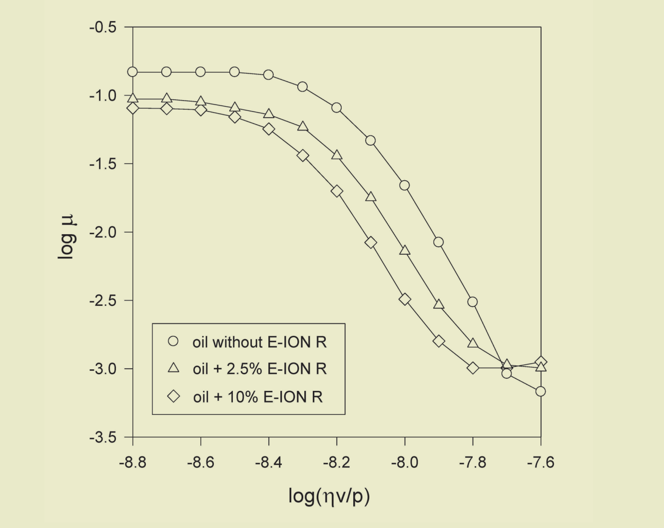
This means that, under identical exploitation conditions, the presence of this lubricity additive is going to reduce friction; the coefficient of friction in the film lubrication regime being about two orders of magnitude lower than that in the boundary lubrication regime. The concentration dependence of the coefficient of friction is related to fractional surface coverage by adsorbed additive, which is concentration-dependent. From a thermodynamic viewpoint, at a given temperature, the degree of surface coverage should increase with increasing concentration of E-ION R in fuel. This has been confirmed experimentally. However, the influence of temperature on friction coefficient is not that straightforward. The reason is that there are both thermodynamic and kinetic factors involved. Thus, an increase in temperature results in higher solubility of E-ION R in fuel, and therefore, the degree of surface coverage at a given additive concentration should decrease, and the friction coefficient, accordingly, increase. In practice, it has been observed that the friction coefficient decreases with temperature. The most feasible explanation of this anomaly is that the adsorbed layer never reaches an equilibrium state in the tribotests conducted. As surfaces are being rubbed against each other, the adsorbed layer keeps constantly being damaged and regenerated. The rate of regeneration is directly proportional to the rate of adsorption, and both increase with temperature. This reasoning shows that evaluations of the degree of surface coverage from the concentration-dependence of friction coefficient may be misleading.

It is important to underline the fact that the experimental conditions chosen for lubricity testing in this study are radically different from those of commonly used "lubricity" standards, such as BOCLE (ASTM D 6078) and HFRR (ASTM D 6079). This may lead to misunderstandings and terminological muddle. For instance, in HFRR, 200 g load is applied to a 6 mm steel ball reciprocating on a steel flat (AISI52100, 650HV). In this case, the Hertzian contact pressure is around 1 GPa, corresponding to a point in the top left end of the Stribeck diagram. This explains why common EP additives, such as molybdenum, phosphate esters and polysulfides, always excel in those tests. In other words, the standard "lubricity" tests do not really test lubricity - they test the EP functionality. If the injection system in a car were continuously exposed to such a stress, its lifetime would have been limited to few days! In reality, the majority of tribosystems in cars are exposed to repeated loading-unloading cycles, with the typical stress range 1 to 10 MPa. The lubricity-enhancing effect of E-ION R is circumscribed to a Hersey number range in which transition from the EHD to the boundary lubrication occurs, and this is outside of the scope of BOCLE and HFRR in their standard setup.

(ii) Mineral oil tests

Mineral oils are common bases for formulation of engine oils, gear lubricants and metalworking lubricants. Needless to say that lubricity is one of the most important essential quality parameters for all those lubricant groups. The experiments show that addition of 10% E-ION R to 100N mineral base oil significantly improves lubricity of the latter (Figure 7).

Figure 7



"Dry" base stocks including severely hydrotreated and hydroisomerized mineral oils and polyalphaolefins are expected to benefit even greater lubricity gain.

(iii) Synergy with conventional EP additives

As already mentioned, ionized vegetable oils are sometimes classified as "soft" EP and antiwear additives. Obviously, it is entirely a matter of convention which pressure is to be called "extreme". As applied to a majority of tribosystems of interest to lubrication engineers, the term "extreme" refers to pressures so high that liquid lubricant is squeezed out from between the rubbing surfaces and a "dry" asperity-asperity contact occurs. Ionized vegetable oils elevate the threshold pressure at which the dry contact will occur but do not provide protection beyond that point. Therefore, for maximum protection under various tribological regimes, it is important that a EP additive be deployed as well in lubricant formulations. Wear preventive characteristics of a few model lubricants formulated with ionized vegetable oils and conventional EP additives were compared using the standard four-ball method, see Table 6. These results show that there is a synergetic effect between ionized vegetable oils and EP additives. A similar conclusion has been arrived at by other authors advocating the use of vegetable oils in automotive lubricants.[15]

TABLE 6) Results of four-ball test carried out on 100N mineral oil treated with two common EP additives and E-ION R friction modifier. The test condition were similar to D2266 (75oC, 1200 rpm, load 50 kg, test duration 1 hour)

Lubricant composition	average scar diameter, mm	observed wear reduction, %
Pure mineral oil 100N	1.09	0
100N + 1% tricresylphosphate	0.74	32
100N + 1% di-tert-butyl polysulfide (TPS 32)	0.71	35
100N + 5% E-ION R	0.61	44
100N + 1% tricresylphosphate + 5% E-ION R	0.52	52
100N + 1% di-tert-butyl polysulfide + 5% E-ION R	0.45	59

There exists evidence that, in many cases, lubricant formulations containing ionized vegetable oils show a significant friction reduction even in the EP domain, where the applied pressure is so high that the organic adsorption film cannot remain intact. This behaviour can be rationalized when taking into account that all real surfaces are rough and it is not really possible to draw sharp borders between boundary, mixed and hydrodynamic regimes. At any given pressure, there will always be some points between the rubbing surfaces (asperity vs asperity) where the boundary lubrication regime takes place, and some points (cavity vs cavity) where the hydrodynamic lubrication regime takes place. Which regime prevails is uncertain. The measured Stribeck curves prove, however, that E-ION friction modifiers significantly expand the range of operational conditions (higher loads, higher temperatures) under which the hydrodynamic lubrication regime is in effect.

The protective surface film formed by ionized vegetable oil can be viewed as a sponge impregnated by base oil, which contains some EP molecules. Within the friction contact, when rubbing surfaces are sliding against each other, such a film undergoes very rapid deformations. The heat generated due to such deformations (proportional to the time-volume integral of the product of film viscosity times velocity gradient squared), in combination with a high pressure, are enough to render EP molecules surface-reactive. As a result, the EP reaction will start before unlubricated contact occurs. This hypothesis is indirectly supported by more detailed investigation of the surface topography of wear patterns obtained in the 4-ball test - in many cases, signs of plastic deformation rather than regular scratches can be noticed.

(iv) Field experience

Lubricity-enhancing effect of ionized oils has been confirmed in field on heavy-duty road transport vehicles by Transports Mommer s.a. in Belgium and by Hindustan Petroleum Corporation in India. The field trials indicated that MIL 2104 C + Mack T7 diesel oils top-treated with E-ION R had better soot control, reduced formation of deposits, leading to increased power output and improved fuel economy (see Figure 8 and Table 7). Oil drain intervals could be doubled. In some tests, MIL 2104 C + Mack T7 oil doped by E-ION R rivals Super High Performance Diesel engine oil (SHPD CCMC D5), see Table 8.

TABLE 7) Field experience proving the beneficial effect of E-ion R friction modifier on fuel economy in heavy-duty commercial vehicles. The achieved annual fuel economy was 5.3%. Data courtesy of Transports Mommer s.a., 69A Route Charlemagne, B-4841 Henri-Chapelle, Belgium.

Data provided by Transports Mommer s.a. Truck type and Belgian registration plate		Regular Commercial Diesel Engine Oil			Fuel-economy engine oil Unimax™ (E-ion s.a.) formulated with 8% E-ion R		
		Mileage	Fuel Cons.	L/100km	Mileage	Fuel Cons.	L/100km
DAF18T	CQQ 107	43806	10349.5	23.63	41332	9294.1	22.49
	DIS 578	29854	7036.7	23.57	40026	10015.9	25.02
	PCM 542	35641	8752.8	24.56	38269	8690.8	22.71
DAF38T	AMW 862	23834	7134.2	29.93	48966	15582.3	31.82
	SEY 249	42769	12681.1	29.65	40919	12783.3	31.24
	SBE 260	46815	14861.8	31.75	51023	13543.3	26.54
	PNC 458	33158	10180.6	30.70	30994	9528.0	30.74
	GGX 472	36806	12261.0	33.31	44320	12948.6	29.22
	JTC 530	41400	12386.3	29.92	44200	12875.8	29.13
	BRI 872	23242	7978.2	34.33	2689	789.0	29.34
	FGQ 141	36543	11426.3	31.27	45747	12671.7	27.70
	DTN 613	45098	13625.3	30.21	58869	16277.6	27.65
	CBW 386	35971	11230.0	31.22	44693	13189.0	29.51
	VAS 655	35645	11177.6	31.36	30727	9569.5	31.14
Total		**510582**	**151081**	**29.59**	**562774**	**157756**	**28.03**

TABLE 8) Physicochemical analysis of engine oils drained after field trials (data courtesy of A.K. Jain, D.V. Prasad, A.K. Bhan, Hindustan Petroleum Corporation, Mumbai, India)

Property	**Engine Oil**					
	MIL 2104 C + Mack T7	MIL 2104 C + Mack T7 with 10% E-ION R				SHPD CCMC D5
		vehicle 1	vehicle 2	vehicle 3	average	
Mileage	22000 km	40000 km				
Visc. 100°C, mm²/s	20.66	17.98	18.49	18.23	18.23	16.88
TBN, mgKOH/g	6.58	8.98	9.77	10.03	9.59	12.7
TAN, mgKOH/g	4.58	3.67	4.09	3.19	3.65	4.14
Hexane insolubles, wt.%	4.66	2.44	2.67	2.84	2.65	1.91
Toluene insolubles, wt.%	3.7	2.18	2.22	2.47	2.29	1.66
Soot, wt.%	4.7	3.03	3.13	2.89	3.02	2.96
Metals, ppm Al Fe Cr Cu Si Pb	 47 224 6 39 69 51	 21 144 7 19 58 34	 24 136 13 27 69 13	 36 159 18 22 56 30	 27 146 13 23 61 26	 19 130 5 27 60 19

Lubricity-enhancing effect of ionized vegetable oils in turbine oil formulations is even more pronounced. As can be seen in Figure 9 and 10, the addition of 10% E-ION R to a turbine oil significantly reduces chatter and eliminates slip-stick.

Figure 9

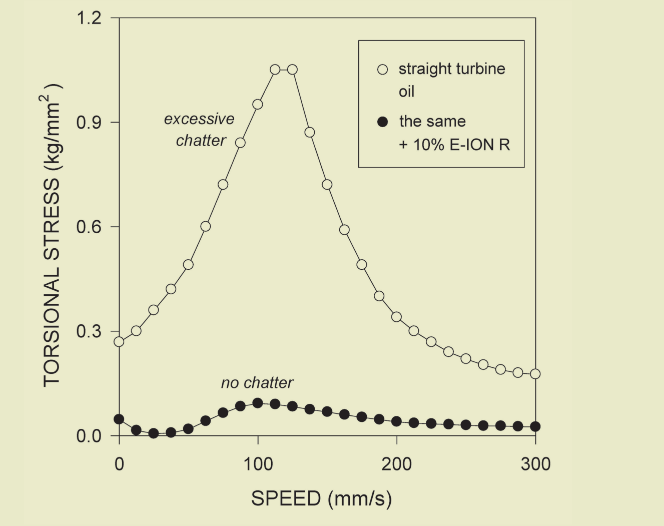
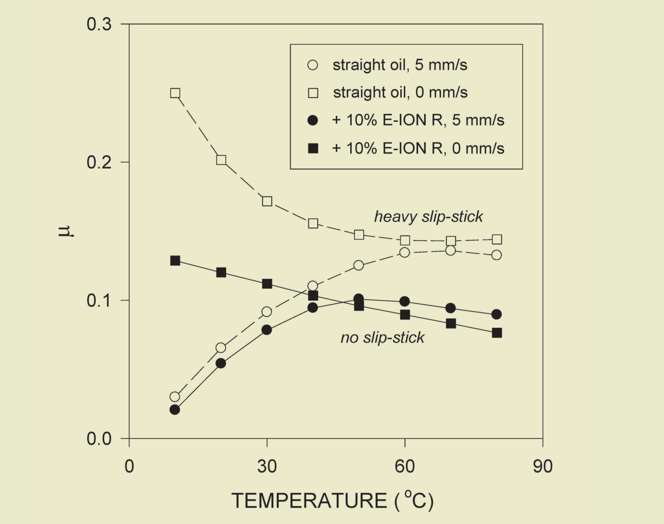


Figure 10



Conclusions

Use of ionized vegetable oils as friction modifiers in lubricants and fuels significantly improves lubricity and antiwear efficiency of the latter. Ionized vegetable oils function as mild antiwear and EP agents. Featuring a unique combination of viscosity and polarity, ionized vegetable oils form sufficiently thick and resilient protective layers by adsorption to rubbing surfaces. Thereby, the onset of boundary lubrication is shifted towards higher loads. Typical tribological scenarios where boundary lubrication, rather than hydrodynamic lubrication, would normally prevail unless lubricity additives are deployed are running engines at dead points, bearings during cold start-up, thrust bearings of vertical turbines during start-up, slideways of high-precision machine tools, railway truck axle box-guides, hydraulic shock absorbers, reduction gears, metalworking operations such as screw cutting, deep drawing, etc.

In all those applications, the use of ionized vegetable oils as lubricity and fatty oiliness additives brings the following benefits: 16

(i) reduced energy/fuel consumption and increased power output;
(ii) enhanced wear protection;
(iii) ability to operate at higher thermal and mechanical loads;
(iv) improved environmental and health safety profile.

In combination with new hydro-processed VHVI and XHVI base oils, as well as emerging Group III+, or "Super-Group III" base stocks produced using the Fischer-Tropsch process, ionized vegetable oils may serve a solid foundation for addressing lubricity challenges in formulation of fuel-economy engine oils, gear lubricants, MWFs, and fuels. Ionized vegetable oils should be used as a complement to but not as a substitute for conventional EP and antiwear additives.

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