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## THIN FILM DEGRADATION TESTING OF LUBRICANTS FOR GAS GENERATOR, DIESEL ENGINE AND HYDRAULIC APPLICATIONS

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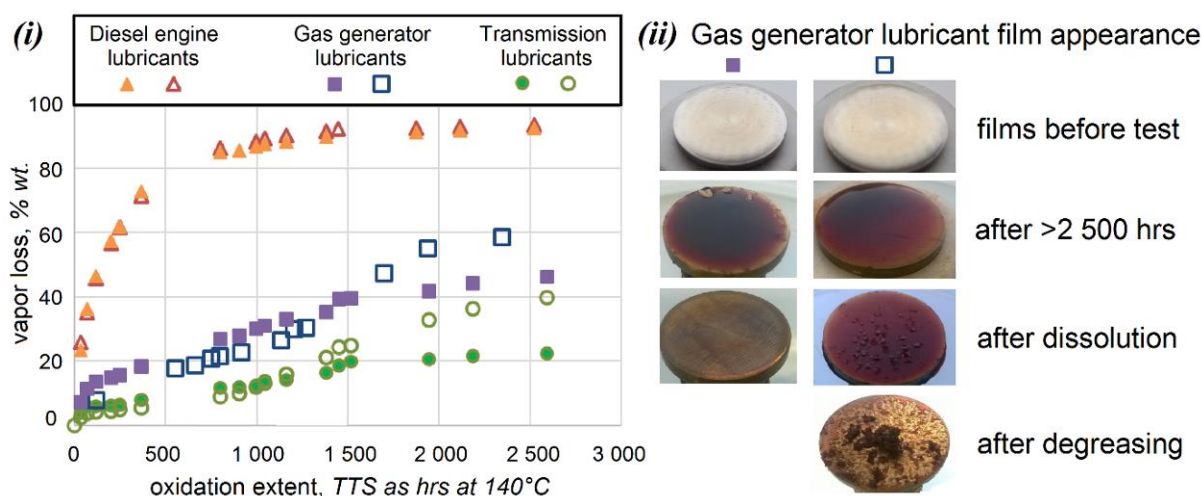
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Lubricant stability is often tested at temperatures above those of normal operating regime to reduce testing durations. Increase in viscosity and acidity frequently constitutes a failing mark, although lubricant still remains liquid. Thus thin film tests gain popularity, because they can show the ability of a lubricant to remain liquid without decomposing into volatiles or polymerizing to solids [1].

Several commercial lubricants were obtained from BM Energy (Latvia) and CJC (Denmark), representing different manufacturers of diesel engine, gas generator and transmission lubricants. Thin film degradation was performed on low carbon steel coupons [1]. Films of initial 500 µm thickness were heated at 130 °C for up to 1815 hrs and then at 140 °C in a Memmert forced draft oven. From 3 to 5 coupons were used for each lubricant and periodically volatile emissions were measured gravimetrically. Typical plots for each lubricant appear in Fig. 1 (i). Time and Temperature Superposition (TTS) was utilized to normalize testing durations at 130 °C to those at 140 °C by applying van't Hoff's rule, i.e. doubling the duration with 10 °C drop in temperature [2], e.g. 1815 hrs at 130 °C into 907.5 hrs at 140 °C.

When suspecting film solidification, the coupon was placed into a beaker with 20 mL of the same fresh lubricant and held for 180 min at 90 °C, stirring it several times. Afterwards the coupon was rinsed with heptane and dried. Most samples eventually produced insoluble residues when degreasing in chloroform for 180 min. Coupons were photographed before and after degreasing or heating.



**Fig. 1.** (i) Volatile emissions from 500 µm lubricant films when heating at 130°C and 140°C, durations normalized by Time and Temperature Superposition (TTS) [2]. (ii) Appearance of representative coupons before and after heating and degreasing (just one sample was degreased) for the two tested gas generator lubricants.

Results show that commercial lubricants produce very different trends of volatile emissions and formation of residues insoluble in fresh oil. Lab observations are likely to relate to field performance.

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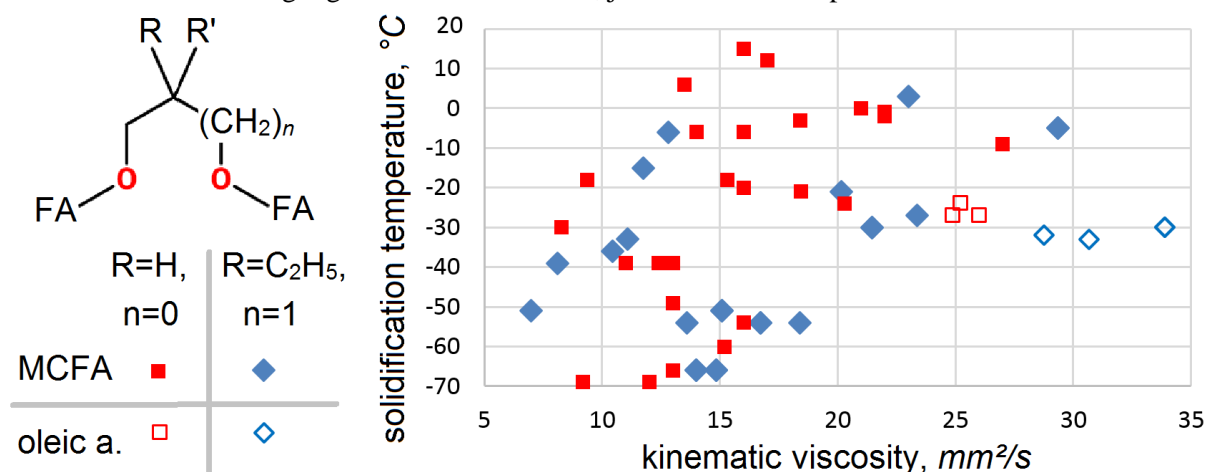
## MONOUNSATURATION EFFECTS ON LOW TEMPERATURE FLUIDITY OF ESTER DERIVATIVES

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Camelina and Crambe crops are potential sources for medium-chain fatty acids (MCFA) from C8 to C14, which represent an important feedstock for specialty oleochemicals. Their oils can also find utilization as lubricants after conversion into esters, if appropriate viscosity is achieved with good fluidity at low temperatures, usually measured as pour point. While MCFA esters might have fluidity problems, cis-double bonds can reduce pour pt. [1] The most abundant fatty acid (FA) in Camelina oil is gondoic (i.e. 11-eicosenoic a.), while Crambe oil contains up to 60 % erucic a. (i.e. 13-docosenoic a.). Initially dibasic esters with MCFA and oleic a. were synthesized, see Fig. 1, using a previously established method [2] with two major types of polyhydric alcohol derivatives: 1) H-containing  $\beta$ -carbon polyol with R=H in Fig. 1 and 2) tertiary  $\beta$ -carbon polyol. Additional branched moieties were also attached as R' for better fluidity. Kinematic viscosities were measured at 40 °C using Cannon-Fenske method (ASTM D455) and in some cases estimated from structural data. Pour pt. was measured under the same thermal cooling regime as in ASTM D97, just in smaller sample sizes.



**Fig. 1.** Molecular structure of synthesized dibasic esters with various FA and R' (left). Their solidification temperatures (measured as pour points, °C) and kinematic viscosities, measured (or predicted) at 40°C (right).

Oleates clearly show that higher mol. weight increases viscosity. Since FA moieties were linear, MCFA esters with viscosities over 20 mm<sup>2</sup>/s could not retain fluidity at -30 °C. However, oleates approached 35 mm<sup>2</sup>/s viscosity with better pour pt. and showed a remarkable tendency to retain fluidity around -30 °C (with tertiary  $\beta$ -carbon) or -25 °C (when R=H) despite variation of R' moieties. Further synthesis with gondoic and erucic esters can give even more flexibility in achieving needed viscosities and lead to broader industrial viability of Camelina and Crambe crops.

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