Lubrication characteristics of wax esters from oils produced by a genetically-enhanced oilseed crop

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ABSTRACT

In this study, purified by a newly developed method Crambe (*Crambe abyssinica*) oil wax esters were mixed with conventional Crambe oil to examine the lubrication characteristics of bio-based wax esters and their blends. The Quartz Crystal Microbalance (QCM) results indicate high thermal stability of wax esters and their mixtures which is further supported by observed small viscosity change with temperature increase. Fourier transform infrared spectroscopy (FTIR) analysis suggested that chemical modifications induced by heating of the Crambe oil with 15 wt% of wax esters are dominated by the stability of wax esters. Tribology tests revealed that adding 15 wt% wax esters facilitates the noticeable friction reduction and lowers the wear produced in the sliding contact at elevated temperatures.

1. Introduction

One of the commonly-used and most effective methods for reducing friction and wear and prolonging the lifetime of mechanical assemblies is introducing a lubricant into the sliding contact [1–4]. Lubricants provide thin-film coverage of the sliding surface thus minimizing their wear and oxidation [5]. While mineral oils possess some desirable lubricating properties at a relatively cheap cost, their use is not appropriate for a range of applications in which the lubricants are used in contact with biological systems, such as the medical or food industry [6, 7]. Moreover, recent demands for increased environmental protection and alternative energy have motivated a large number of research studies focused on the use of vegetable oils as lubricants for tribological applications [1,8–11].

Recent efforts include a detailed analysis of the origin of lubricity in plant-based lubricants [12,13]. By examining the seed oil of *Orychophragmus violaceus*, it was shown that thermal stability and lubricity largely depended on the structure of component estolide molecules [12, 13]. This knowledge suggested a new approach for improving the lubrication characteristics of other plant-based oils, such as castor oil

[13]. However, manipulating the estolide content is a challenging task. Alternative efforts are needed for improving the lubrication characteristics of widely used crops.

Jojoba oil has attracted considerable attention in the lubrication field due to its efficiency in improving viscosity, as well as antifoaming and anti-corrosion characteristics of oil formulations [14,15]. Improved lubrication characteristics of the jojoba oil are attributed to the oil's composition, which is almost completely made of wax esters [16]. Wax esters consist of one fatty acid and one fatty alcohol linked to each other through an ester bond rather than fatty acids to a glycerol backbone as in conventional triacylglycerol (TAG)-containing vegetable oils [17]. Wax esters are of high interest for many non-food applications as components of cosmetics and other personal care products. Wax esters also exhibit favorable lubricating properties, however, their significantly higher price as compared to conventional plant oils limits the use of wax esters as lubricants and protective coatings [18,19]. However, often inconsistent supplies of wax esters from jojoba, production of which is limited to more arid environments, highlights an existing need for expanded production of bio-based wax esters [20]. Therefore, efforts have been pursued to produce wax esters in genetically engineered oil crops and

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several successful attempts have been reported [21–23]. Search for an alternative source of wax esters brought attention to Crambe oil. The high content of the monounsaturated erucic acid (docosa-13-enoic acid; 22:1Δ13) acids and the low levels of di- and tri-unsaturated fatty acids in Crambe oils render it interesting for some industrial applications, including lubrication [24]. And now transgenic Crambe plants have been developed that produce seeds with various amounts of wax esters mixed with a larger proportion of TAG [21].

Here, we explore the possibility of the wax esters extraction from the transgenic Crambe oil. We report a new method to isolate the wax esters from such oils and analyze their lubrication characteristics at different temperature regimes in mixtures with conventional Crambe oil. We demonstrate how the addition of these wax esters added to Crambe oil can be used to improve the thermal stability of the oil and to reduce the friction and wear of the oil-lubricated steel surfaces in an elevated temperature regime. Our results bring new knowledge to understanding the lubrication characteristics of the wax esters that can be extracted and purified from genetically-modified oilseed crop sources to use in a wide variety of blends of bio-friendly lubricants.

2. Experimental Procedure

Wax esters isolation: Crambe seeds from plants expressing jojoba fatty acid reductase and wax synthase containing about 20 wt% wax esters in their oil [21] were harvested from field trials and the oil was extracted with heptane from crushed seeds. After evaporation of the heptane with a rotary evaporator, the remaining oil (~500 mL) was mixed with 2 vol (~1 L) of 0.1 M sodium hydroxide in water-free methanol and was agitated for 20 h at room temperature (22 °C). After the addition of two volumes (~1 L) of 1.2 M HCl and 10 wt% NaCl in aqueous solution to the fraction, the phases were allowed to separate overnight and the oil fraction was collected. Separation of the oil fraction on silica TLC in heptane/diethyl ether/acetic acid (85:15:1 by vol.) showed that all TAG were converted to methyl esters whereas the wax esters remained intact. The oil fraction was distilled in a short path distillation equipment at 0.1 mbar pressure. Methyl esters, free fatty acids, and any free alcohols were collected in fractions <300 °C and the wax ester fraction was collected between 300 and 320 °C.

Isolated wax esters were partly solid in room temperature (22 °C) and centrifuged at 20 000×g at 18 °C for 30 min and the liquid fraction was decanted. The solid fraction made up approximately 10% of the total wax ester volume. The component composition of the wax esters was analyzed following methanolysis. Aliquots of the solid and liquid fractions were methylated with 2 wt% H_2SO_4 in water-free methanol at 90 °C for 60 min. The fatty acid methyl esters and free alcohols were extracted into heptane and analyzed by gas chromatography (GC) equipped with a flame ionization detector (FID) detector for peak quantification and with gas chromatography/mass spectrometry (GC/MS) for peak identification. For FID detection, the methyl esters and fatty alcohols were separated and analyzed by GC-FID (Shimadzu CG-17A) on a 50 m DB-WAX column. GC/MS analyses were performed on an Agilent 5975 mass-selective detector coupled to an Agilent 6890 gas chromatograph equipped with an HP-5MS column (30 m × 0.25 mm i. d., and 0.25 μm film thickness; J&W Scientific, USA). Helium was used as carrier gas at a constant flow of 1 mL/min corresponding to the linear velocity of 36 cm/s. The oven was programmed at 60 °C for 1 min, then to 220 °C at a rate of 20 °C/min, held for 2 min, and finally to 280 °C at 10 °C/min, held for 30 min.

Oil sample preparation: The triacylglycerol-based oils used in the studied were cold-pressed from non-transgenic Crambe seeds. The oil mixing with wax esters was performed as follows: the purified liquid wax ester fraction was mixed with the conventional Crambe oil at 5, 10, 15 and 20 wt% concentration by volume (labeled 5%, 10%, 15%, and 20% correspondingly). Notably, once prepared, the samples showed no signs of degradation of sedimentation even after one year of sample storage.

Quartz crystal microbalance (QCM) tests: Analysis of the viscosity changes during the heating of the oils was performed using a custom-made QCM setup [5]. QCM allows in-situ monitoring of the changes in the solid/liquid contact in a non-destructive way [25–28]. Chromium-coated AT-cut crystals (1 in. in diameter) with 5 MHz base resonant frequency purchased from Fil-Tech were used in this study. After fixing the crystal with electrical contacts running at the bottom of the crystal and being insulated from any possible contact with oil, 5 ccs of oil were introduced on the top side of the crystal. The readings of the resonant frequency of the QCM oscillations were stabilized after adding the oil for 60 min at room temperature (22 °C). Once the frequency reaches a stable regime, the QCM setup was placed for heating analysis in a box furnace (Thermo Fisher). The QCM signals were gathered by SRS QCM200 during stabilizing and heating regimes. Viscosity changes in the oil were calculated based on the classical understanding of the QCM frequency changes in a viscous medium:

$$\Delta f = - f_u^{1.5} \left(\frac{\rho_L \eta_L}{\pi \rho_q \mu_q} \right)^{0.5} \tag{1}$$

where f_u is the frequency of oscillation of unloaded crystal, ρ_q is the density of quarts (2.648 g cm^{-3}), μ_q is the shear modulus of quartz (2.947 × 10^{11} g cm^{-1} S^{-2}), ρ_L is the density of liquid in the contact with the electrode which was measured separately by using a precise pipet and balance with 0.0001 g accuracy, and η_L is the viscosity of the liquid in contact with the electrode.

Tribology tests: To evaluate the lubrication efficiency of the oils, tribology tests were performed using the Anton Paar macroscale ball-on-disk tribometer. For this, 6 mm in diameter stainless steel (440C) ball was sliding against the mirror-polished (roughness ~ 20–30 nm) 440C stainless steel flat with contact being lubricated with several drops of oil. The substrates and the counter bodies before the tests were cleaned with acetone followed by isopropanol. The linear reciprocating mode was used for all the tests (2 mm running distance at 1 Hz frequency). The tests were performed at a maximum Hertzian contact pressure of 1.1 GPa, indicating the boundary lubrication regime, the regime when sliding surfaces being close to each other form only a thin oil-based protective layer in between. The heating stage tribometer assembly was used to run the tests at elevated temperatures in the range of 25–200 °C with ±1 °C temperature accuracy. To ensure the reproducibility of the results, each test was repeated at least three times.

Wear characterization: To characterize the samples after the tests, the excess of oil was removed from the samples. After this, the samples were rinsed in acetone followed by isopropanol and dried under a dry nitrogen flow. The wear volume of wear scar on the pin side was calculated based on the ASTM G99 standard [29]:

$$V = \left(\frac{\pi h}{6} \right) \left(\frac{3d^2}{4} + h^2 \right) \tag{2}$$

where d is the wear scar diameter, r is the radius of the ball and

$$h = r - \sqrt{r^2 - \frac{d^2}{4}} \tag{3}$$

The optical images of the wear tracks were acquired using a Zeiss optical microscope. Profilometry tests were performed using Veeco Dektak 150 profiler. The micrographs and Energy Dispersive Spectroscopy (EDS) mapping were performed using the Hitachi Tabletop ESEM TM3030Plus (table-top SEM) equipped with EDS. The oxidation of the wear tracks was further characterized by Raman analysis performed using Nicolet Almega XR Dispersive Raman spectrometer with a green laser (wavelength of 534 nm). Fourier transformation infrared spectrometer (FTIR) analysis was performed with the FTIR Nicolet 6700 with a 1000−4000 cm^{-1} spectral range.

3. Results and discussion

3.1. Purification of wax esters from the seed oil of genetically modified Crambe

Crambe seed oil from plants expressing jojoba fatty acid reductase and wax synthase, containing about 20% wax esters in their oil [21] were subjected to mild methylation conditions (see Experimental Procedure) leading to complete methylation of the TAG, leaving the more methylation-resistant wax esters intact (Fig. 1). Since long-chain TAG and long-chain wax esters have similar boiling points and the fatty acid methyl esters are much more volatile than wax esters, this greatly simplified the separation of methyl esters and wax esters by distillation, yielding a near pure wax ester fraction (Fig. 1).

The wax ester fraction was partly solid at room temperature (22 °C), and the solid part was separated from the liquid part with centrifugation. GC analyses of the fatty acids and fatty alcohols after methylation showed that solid and liquid fraction had similar fatty acid and alcohol composition but the solid fraction was enriched with saturated fatty acids and alcohols longer than 20 carbons (Fig. 2). Wax esters with these carbon chains act probably as a core for crystallization. The Crambe wax esters were found to be similar to jojoba oil wax esters with regard to their high content of erucic alcohols and eicosaenoic (20:1Δ11) and erucic (22:1Δ13) fatty acids. However, jojoba wax esters have much higher levels of eicosaenoic alcohols and much less of erucic fatty acids than the Crambe wax esters [30].

3.2. Tribological analysis

The purified liquid wax ester fractions were mixed in different concentrations with the Crambe oil. The basic characteristics and stability of the oil mixtures were analyzed using the QCM setup (Fig. 3), allowing in-situ monitoring of the dynamics of the oil/surface interactions [31]. Fig. 3a illustrates a schematic diagram of the QCM setup that allows measuring the changes in the oil density and viscosity through in-situ monitoring of the changes in the resonant frequency and mechanical resistance of the QCM oscillations. Fig. 3b shows the QCM set up before and after it is submerged in the Crambe oil and the mixtures of Crambe oil with wax esters. The tests were conducted in a furnace with an accuracy of ±1 °C and at 0.5 °C/min heating rate from room temperature (22 °C) up to 100 °C. After cooling down the furnace to room temperature (22 °C), the heating process was repeated for the second time (Fig. S1) to analyze how thermal cycling affected the physical
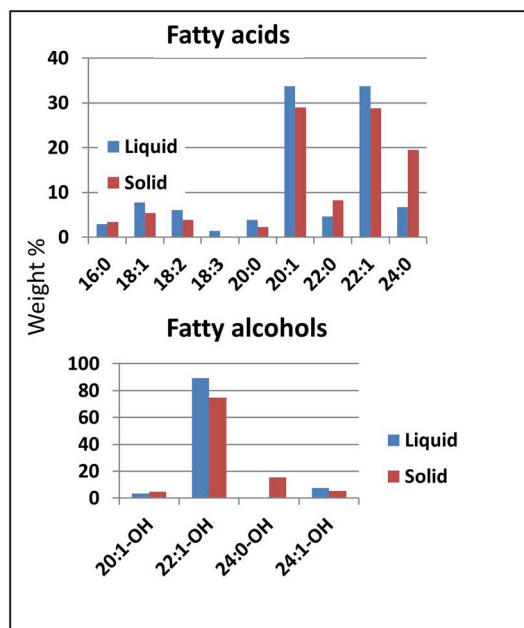
Fig. 2. Fatty acid and fatty alcohol composition of purified wax esters from the seed oil of genetically engineered Crambe plants. Liquid and solid denote fractions of liquid and solid at 18 °C, respectively.

characteristics of the mixed oils.

The changes in the QCM frequency (and mechanical resistance) as a function of temperature for the first and the second heating cycle are shown in Fig. 3c and d (Fig. 3e and f) respectively. As shown in Fig. 3c, the largest change in the frequency during heating belonged to the pure Crambe oil, suggesting that the oil significantly loses in its viscosity and its lubrication characteristics of the oil may suffer at elevated temperature. Adding the wax esters to the oil resulted in a decrease in the delta frequency vs temperature slope, with a higher decrease for a higher amount of wax esters added to the mixture. This behavior was also observed for the second heating cycle (Fig. 3d). The lower sensitivity of the oil + wax esters mixtures to the temperature changes suggests their higher stability as a lubricant during heating. A similar trend was observed for the mechanical resistance of the QCM oscillations (Fig. 3e and f) further supporting the slower viscosity change rates for the
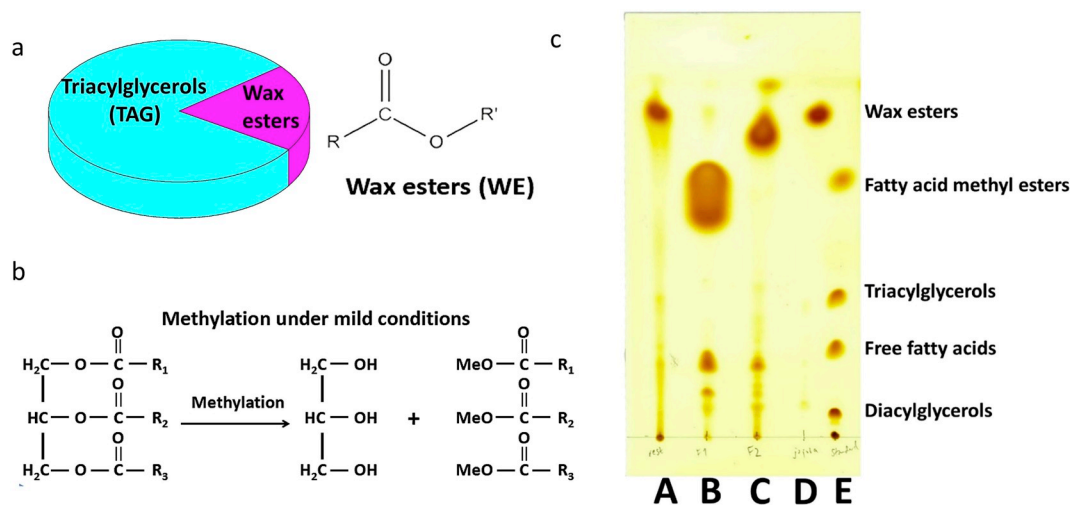
Fig. 1. (a) Schematic diagram shows the concentration of the wax esters in the Crambe oil and their chemical structure. (b) Methylation of modified Crambe oil to purify the wax esters (c) TLC separation of lipid fractions from methylated Crambe oil from genetically modified plants producing wax esters after short path distillation. A) Residue B) Fraction <300 °C, C) Fraction 300–320 °C, D) Jojoba oil as a standard, E) Lipid standards.
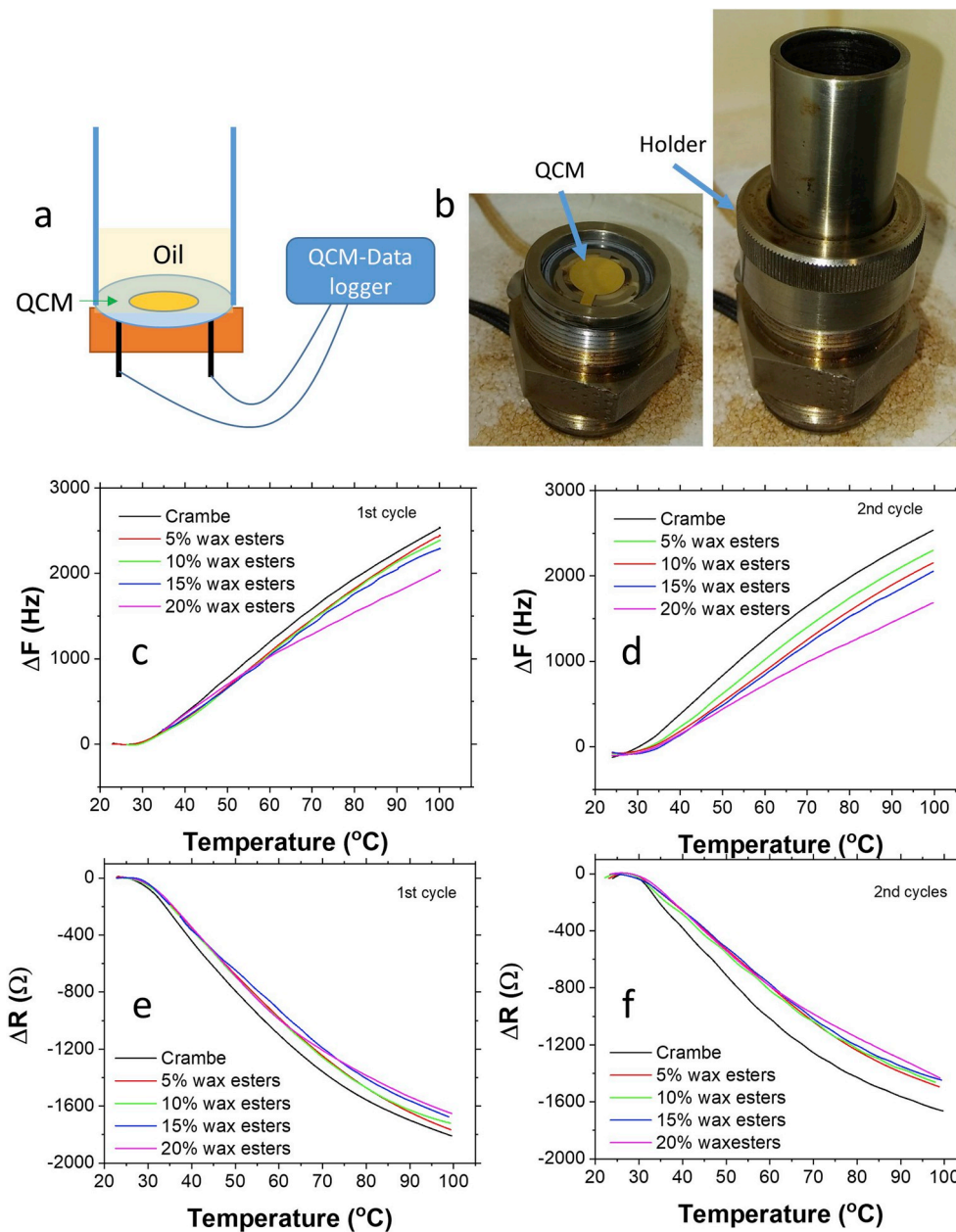
Fig. 3. (a) Schematic diagram of the QCM setup. (b) QCM after the cleaning fixed in the holder inside the furnace right before measurements. (c) & (d) Delta frequency versus increasing temperature between 25 and 100 °C with 1 °C/min ramping up for the first cycle of tests and the second cycle of tests respectively. (e) & (f) Delta resistivity of QCM which gathered simultaneously with Delta frequency.

mixtures.

The mixtures of the oil with wax esters were further analyzed for their tribological performance in the macroscale pin-on-disk tests at 25 and 100 °C. Fig. 4 illustrates the schematic diagram of the tribometer (Fig. 4a) and produced wear scar (Fig. 4b). The frictional behavior of the conventional Crambe oil and Crambe oil with wax esters mixtures at 25 °C is shown in Fig. 4c. At room temperature (22 °C), wax esters did not show dominant improvement in the frictional behavior of base oil, and the COF values for all oils remained in a similar range. However, at 100 °C (Fig. 4d), the positive effect of wax esters was obvious. Wax esters in all concentrations had better frictional behavior than the Crambe oil at 100 °C. Corresponding wear scar analysis for short-duration tests (Fig. S2) is presented in the Supplementary Information. To further explore the lubrication characteristics of the oils at elevated temperature, we performed the long duration tribology tests at 100 °C for 100000 cycles (Fig. 4e). The durability tests indicated that adding 15%

of wax esters to the oil resulted in the most pronounced effect on the COF values. Optical microscopy analysis indicates no significant changes in the width of the wear tracks, however, the profilometry tests suggest more shallow nature of the wear tracks lubricated with the addition of the wax esters (Fig. S3). The calculated wear volume based on the profilometry tests revealed that lubrication with 15% wax esters results in a 55% lower wear rate than the lubrication with pure Crambe oil (Fig. 4f). Since 15% of wax esters show the best frictional and wear resistance behavior, further performance analysis and characterization were focused on the Crambe oil with 15% wax esters.

To further analyze the origin of the surface protection of oils, we performed the energy-dispersive x-ray spectroscopy (EDS) mapping (Fig. 5) of the wear tracks formed on a flat surface and on the counter body (ball). Fig. 5a and b shows surface contact areas of Crambe oil with 15% Wax esters with corresponding elemental maps. The wear track width of 15% wax esters is slightly narrower than the Crambe oil. Also,
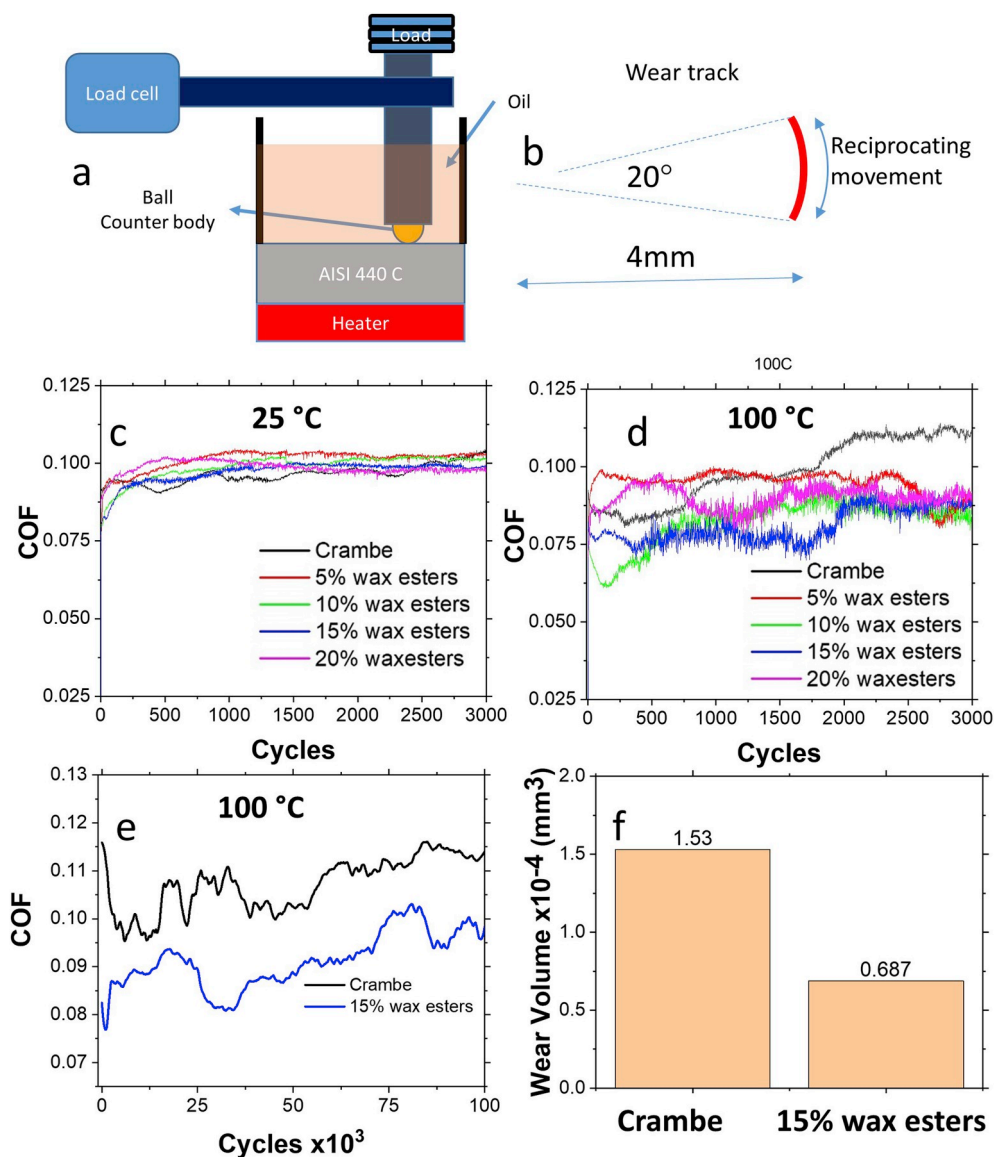
Fig. 4. (a) Schematic diagram of tribometer as well as produced reciprocating wear track. (b) Coefficient of friction behavior of different oils as a function of wax esters additive at (c) 25 °C and (d) 100 °C. (e) Durability tribological tests for 100k cycles as a function of wax esters additive. (f) Calculated wear volume is based on the wear track profiler data (Fig. S3).

the wear scratches of the Crambe oil (Fig. 5a and c) are deep enough to make some grooves. However, for the 15% wax esters, the roughness of the wear scars is smoother than the pure Crambe oil and includes much shallower micro-grooves (Fig. 5b and d) which agrees with the data from the profilometry tests (Fig. S3).

The presence of oxygen inside the wear track for pure Crambe oil is well distributed over the deep micro groves (Fig. 5a and c). However, a 15% wax ester mixture shows the patchy presence of oxygen (Fig. 5b and d). The EDS analysis of the entire surface reveals that the oxygen percentage was 3.73 wt% and 3.52 wt% for Crambe oil and Crambe oil with 15% wax esters, respectively (Fig. S4). These values over the counter body for pure Crambe oil and with 15% wax esters are 2.74 wt% and 1.54 wt%, respectively (Fig. S5) indicating less oxidation with wax esters. The oxygen mapping and elemental analysis of the wear track show the better surface protection of the sliding steel interfaces against oxidation when lubricated with the 15% wax esters mixture in comparison to the pure Crambe oil. The carbon EDS mapping of the surfaces after the test in the Crambe oil indicates the formation of carbon mostly only inside the wear track, thus indicating some initial signs of the oil combusting and sludge formation (Fig. 5a and c). In contrast, for the

15% wax esters mixture, carbon is more evenly distributed over the inside and outside wear track areas (Fig. 5b and d) and the overall amount of carbon inside the wear track in case of the 15% wax esters is much lower (29.3 at% in contrast to 46.6 at% in case of the Crambe oil alone).

We further explored the lubrication characteristics of the oils at temperatures >100 °C (Fig. 6). Fig. 6a summarizes the coefficient of friction (COF) variations as a function of temperature for pure Crambe oil, pure wax esters, and Crambe oil + 15 wt% wax esters. For these studies, the tribology tests were conducted from room temperature (22 °C) up to 200 °C while heating the oil and sliding steel surfaces at a rate of 3 °C/min. Samples consisting of 15% wax esters and pure wax esters had reduced COF starting from around 75 °C, while the COF for conventional Crambe oil remained almost unchanged. Therefore, this reduction in the COF for 15% wax esters at elevated temperatures is attributed to the changes in the wax esters lubrication properties, which become dominant over conventional Crambe oil after 75 °C. To test the concept that the wax esters affect the physical properties of mixed oil at high temperatures, QCM measurements were made using the same heating rate as for the tribology tests (3 °C/min). QCM delta frequency
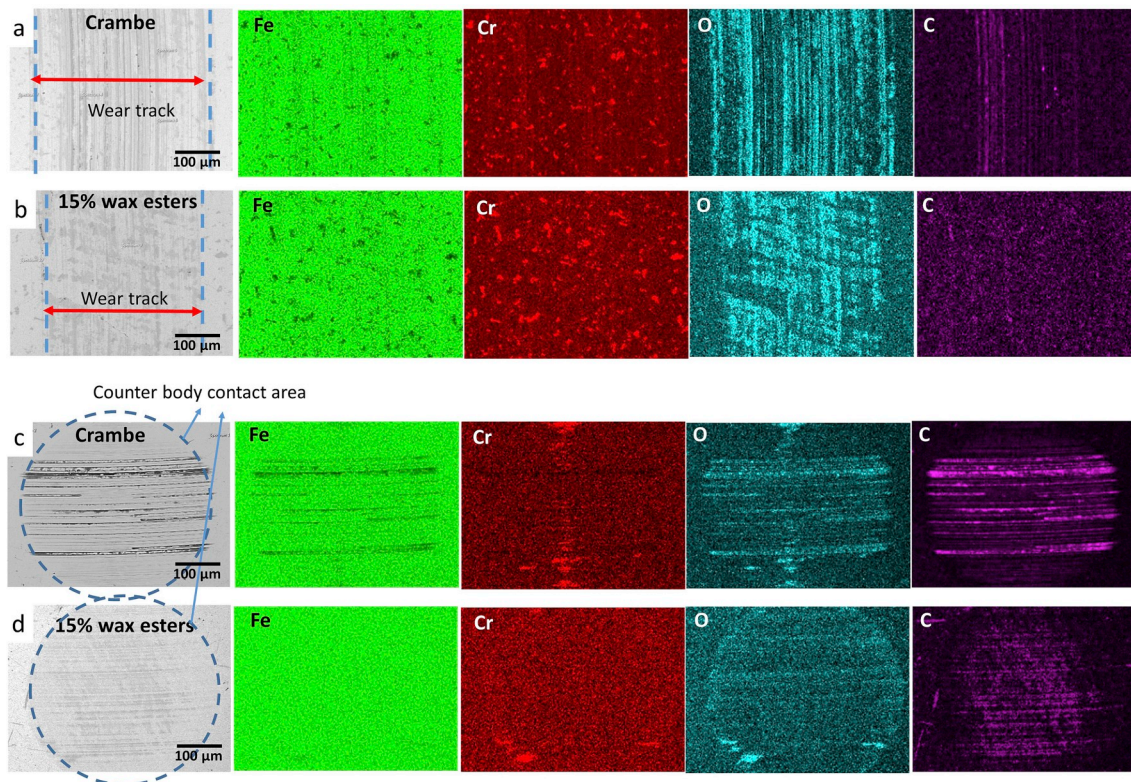
Fig. 5. (a) Micrographs and EDS mapping of wear track's surface area as well as the correspondent counter bodies for (a) & (c) Crambe oil and (b) & (d) Crambe oil + 15% wax esters respectively.
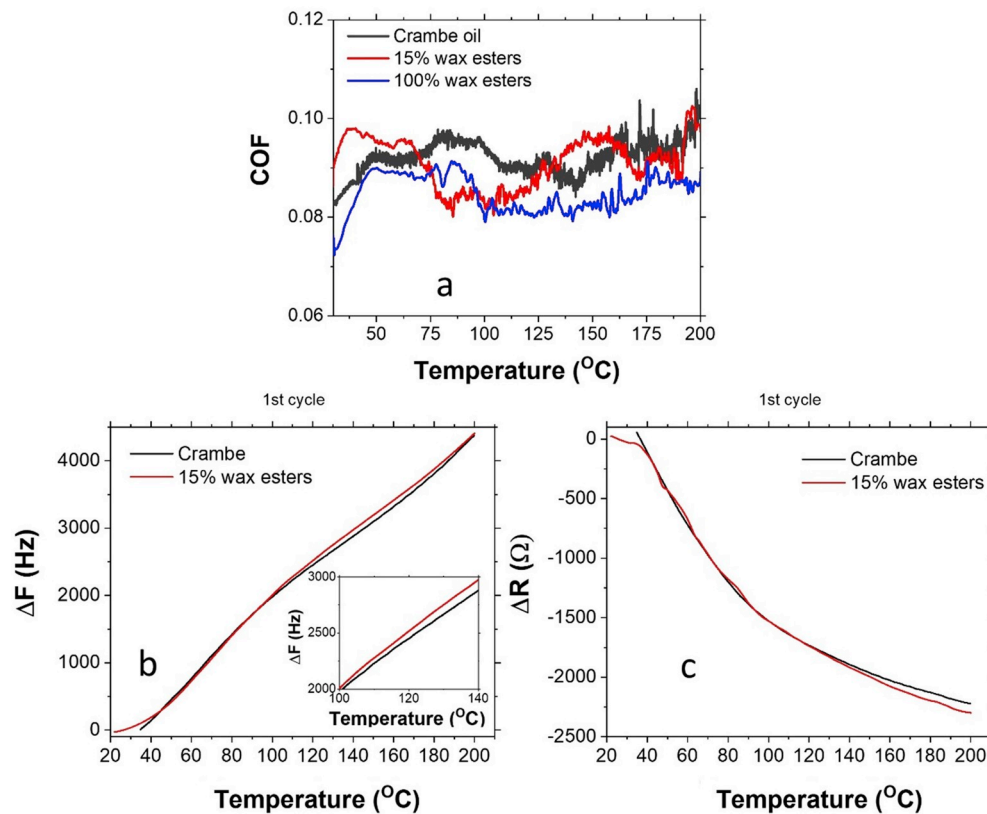

Fig. 6. (a) Variation of the coefficient of friction as a function of rising temperature from 25 to 200 °C with a 3 C/min rate. (b) & (c) QCMs delta frequency and delta resistivity of Crambe and Crambe oil + 15% wax esters during raising the temperature with the same heating rate as the tribology test was done.

analysis is shown in Fig. 6b. At low-temperature regime (25–90 °C), the Crambe oil had higher values than the one with 15% wax esters. However, at moderate temperatures (100 °C), a transition occurred and delta frequency of 15% wax esters became higher than that for Crambe oil. This observation suggests better adaptability of the wax-esters to higher temperature regimes thus enabling steel surface protection. Also, delta mechanical resistance of the QCM oscillations showed the same trend: 15% of wax esters experienced a reduction in the mechanical resistance of the QCM oscillations after 100 °C (Fig. 6c).

Slight offset in the temperature transitions observed for the COF values and the QCM oscillations changes (75 °C in contrast to 100 °C) can be attributed to the nature of the macroscale tribology tests which lead to local heating events during the sliding of the surfaces.

A more detailed analysis of the lubrication characteristics of the oils (Fig. 7) at different elevated temperatures indicated that improvements in the COF and wear values are observed at ≤200 °C. Fig. 7a–e illustrate COF of Crambe oil with 15% wax esters from 25 °C up to 200 °C. At room temperature (22 °C), both COFs demonstrated similar values (Fig. 7a). After raising temperature to the transition temperature around 75 °C and up to 150 °C, 15% wax esters showed a reduction in friction in

comparison to the pure Crambe oil. During further increase in temperature to 200 °C, both oils demonstrate signs of oil degradation and polymerization resulting in unstable COF behavior [32] (Fig. 7e). These results were in agreement with the QCM analysis indicating that at 200 °C wax esters are expected to experience initial signs of the degradation leading to deterioration in their lubrication characteristics. Fig. 7f shows the calculated wear volume based on the contact area over the counter body (Fig. S6).

To understand the nature of the observed modifications in the friction and wear reduction characteristics of oils, we performed the FTIR analysis of the Crambe oil with 15% wax esters, conventional Crambe oil, and pure wax esters (Fig. 8). The behavior of 15% wax esters was almost identical to pure wax esters which indicates the dominance of wax esters over the base Crambe oil at high temperature. Specifically, in contrast to pure Crambe oil, both 15 wt% wax esters and 100 wt% wax esters samples demonstrate no changes in CH bond stretching upon annealing, which suggests that wax esters help to limit the oil degradation upon heating. Slight increase in ester (C=O) peak at 1739 cm^{-1} for 15 wt% wax esters at 200 °C indicates the effect of the ester groups on limiting the oxidation process [33]. Both Crambe and wax esters oils
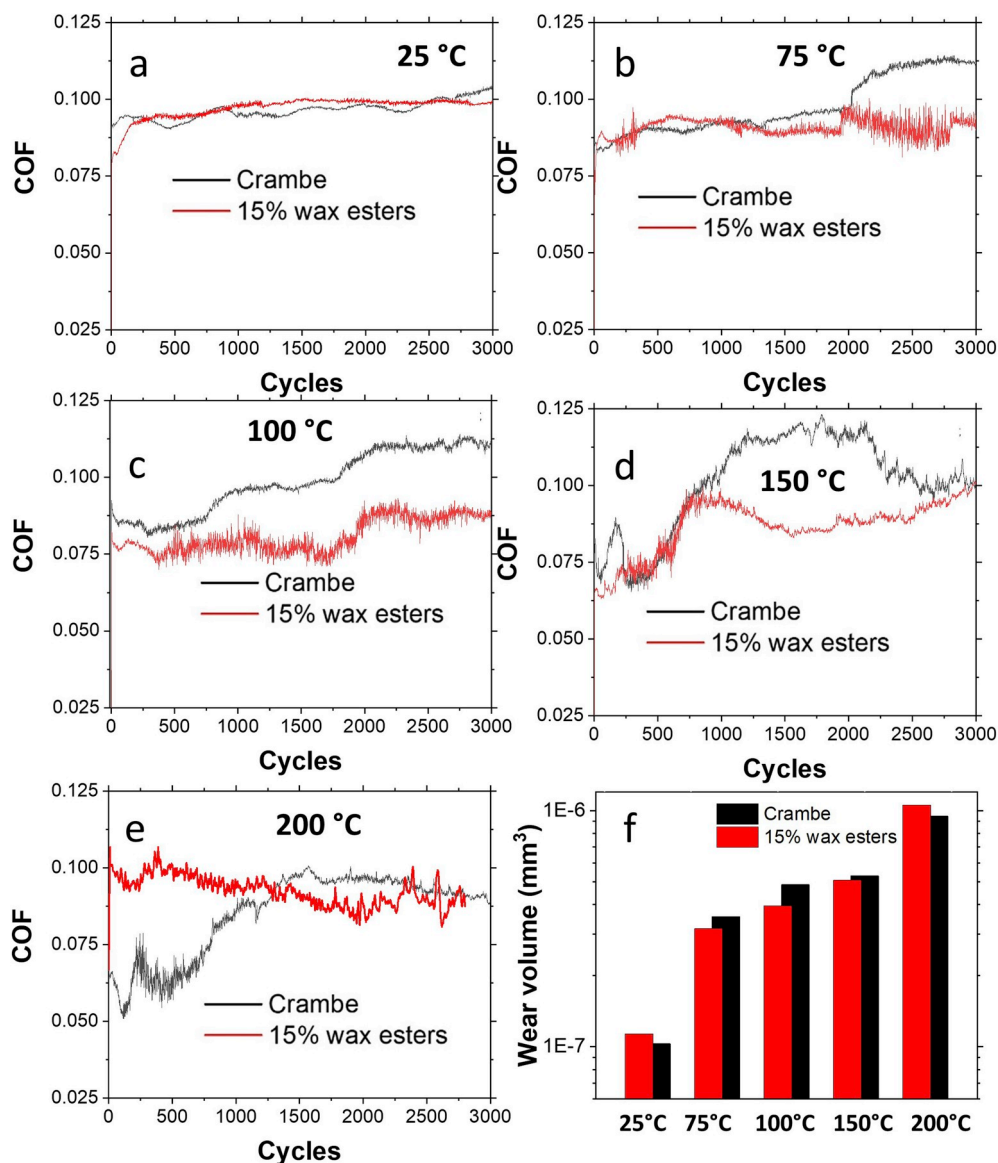

Fig. 7. Coefficient of friction behavior for Crambe oil and Crambe oil with 15 wt% wax esters at (a) 25 °C, (b) 75 °C, (c) 100 °C, (d) 150 °C and (e) 200 °C. Wear volumes were calculated based on the contact area surface area (Fig. S6).
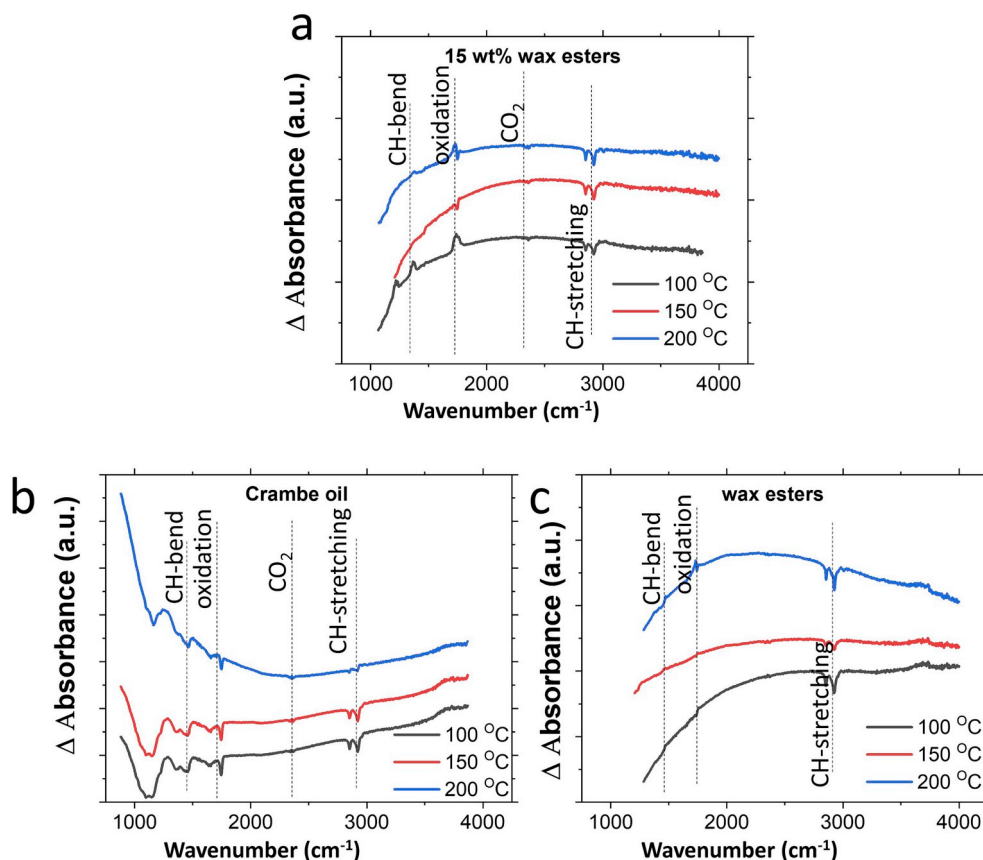
Fig. 8. FTIR spectra of 15% wax esters (a), Crambe oil (b) and pure wax esters (c) at 100, 150 and 200 °C. The spectra are gathered against the same oil from 25 °C.

show CH bond stretching (Fig. 8). At elevated temperature (200 °C), Crambe oil loses its CH stretching and starts to degrade. In contrast, wax esters and their mixture with the Crambe oil still demonstrates the CH stretching. This means wax esters are more stable than pure Crambe oil at 200 °C (Fig. 8c). Thus, the presence of wax esters in the mix oil promotes the existence of CH stretching, providing better and more stable lubrication at elevated temperatures. This finding explains why Crambe oil with 15% of wax esters is more stable than the pure Crambe oil for high-temperature regimes (Fig. 7).

4. Conclusions

Crambe abyssinica has been previously genetically engineered to produce about 20% long-chain wax esters of total seed oil [21]. Here we present a simple method to purify these wax esters by mild methylation of the oil combined with short path distillation. As a side product, fatty acid methyl esters are obtained, which are useful for non-food applications such as biodiesel.

Crambe oil is a biodegradable oil and demonstrates high application potential in the area of plant-based lubricants from renewable resources. Here we demonstrate that the lubrication properties of conventional Crambe oil can be further improved by adding wax esters that were purified from the oil of genetically modified Crambe seeds. Adding wax esters to Crambe oil to a concentration of 15 wt% improves the temperature stability of the oil which has been confirmed by tribology, QCM, and FTIR analysis. Tribology tests confirmed better wear resistance of wax esters and Crambe oil-wax ester blends than that of the base Crambe oil. Detailed tribological analysis indicated the optimum concentration of the wax esters, 15 wt%, results in 55% lower wear volume than for the Crambe oil at 100 °C. Also, adding wax esters to Crambe oil conferred enhanced oxidative resistance, as demonstrated by the EDS mapping.

QCM study of the Crambe oil with wax esters revealed that at a temperature above 100 °C the wax esters properties dominate over the Crambe oil properties. The lower sensitivity of the wax esters to the changes in the suggests their higher stability as a lubricant during heating. The mechanical resistance measurements performed using the QCM further support this observation by indicated less temperature-induced viscosity changes for the wax esters with Crambe oil mixtures.

FTIR analysis suggested that observed modifications induced by heating of the Crambe oil with 15 wt% of wax esters are dominated by the wax esters stability. Both Crambe and wax esters oils show CH bond stretching. At elevated temperature (200 °C), Crambe oil loses its CH stretching and starts to degrade. In contrast, wax esters and their mixture with the Crambe oil still demonstrates the CH stretching. This means wax esters are more stable than pure Crambe oil at 200 °C. Thus, the presence of wax esters in the mix oil promotes the existence of CH stretching, providing better and more stable lubrication at elevated temperatures.

Our results indicate that oil produced from genetically engineered Crambe with ≥15 wt% wax esters in its oil will have an added value in lubrication applications without further processing or by diluting with conventional Crambe oil to a concentration of 15 wt% wax esters.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Asghar Shirani: Formal analysis. **Tasha Joy:** Formal analysis. **Edgar B. Cahoon:** Writing - original draft. **Kent Chapman:** Writing -

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi. org/10.1016/j.triboint.2020.106234.

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