



RECENT ADVANCES IN BIOLUBRICANT TECHNOLOGIES

Dr Raj Shah, Clark Ye and William Chen discuss the recent advances in biolubricants and where the future direction may be taking us

Improvements to bio-lubricant characteristics often rely on modifications to their inherent chemical structure. The synthesis of these novel bio-lubricants is derived from innovative chemically synthesised bio-oil feedstocks.

Over the years, estolides, a type of bio-oil, have been gaining attention. This is due to their ability to be tailored by oligomeric length and functional groups to achieve desired characteristics in viscosity and oxidation resistance [6].

In addition to their high potential in the bio-lubricant industry, estolides are known for their incredible biodegradation and non-toxicity.

Recently, improvements have been made to estolides, including novel synthesis methods using a mesoporous aluminosilicate [7]. Natural, plant-based oils or natural fatty acids are often used as bio-lubricants or in the synthesis of estolides.

Substantial research has been conducted on the properties of efficient bio-lubricants. The research demonstrated a strong connection between erucic acid and lubrication efficiency [8]. The greater the content of erucic acid found in the plant-based oil, the greater the lubrication efficiency of the oil.

Another example of a bio-lubricant that’s shown to improve lubricant efficiency is wax esters. This class of esters is also mainly derived from plants.

In both cases, the isolation of the responsible fatty acid or ester led scientists to push the limits of production through novel genetic engineering.

Quite recently, scientists have developed possible methods to boost both erucic acid concentrations and wax ester production through genetic modification of both leaves and the seed through agrobacterium [9], [10].

Estolides

Estolides are produced through the formation of a carbocation at the site of unsaturation from a carboxylic acid and a fatty acid [11]. A positive charge is formed at the site of reaction. This then forms an ester linkage through nucleophilic addition with another fatty acid.

These linkages considerably strengthen the estolides’ resistance to hydrolysis, which is a desirable trait in bio-lubricants [12].

This characteristic allows the bio-lubricant to resist degradation under high temperatures and the presence of water.

Over the years, researchers have developed unique methods for the mass production of estolides. Some examples include synthesizing estolides from oleins (a class of unsaturated hydrocarbons) obtained from sunflower oil, rather than fatty acids, via a sulphuric acid-catalysed synthesis [13], [14].

This experiment objective focuses on transforming low-value byproducts into high-value estolides, while observing the effect of reaction time on the estolide kinematic viscosity.

Controlling the kinematic viscosity allows the use of estolides in a variety of lubrication applications.

In an experiment, 100 g of olein samples were equilibrated to 50°C and catalysed with concentrated sulphuric acid.

The samples were purified and analysed.

The results show a strong, direct correlation with the molecular weight of the estolides formed.

Molecular weight directly affects the estolide’s viscosity, which leads to the conclusion that the degree of oligomerisation (the process of producing estolides) can be controlled through reaction length [14].

As seen in Figure 2, the extent of the reaction had the most significant impact during the first three hours and adversely affected the kinematic viscosity between 12 and 24 hours.

However, it’s not enough to consider the kinematic viscosity.

The data in Figure 1 show an increase in kinetic viscosity with reaction extent. This suggests that viscosity can be controlled by reaction extent. However, the desirable characteristic of bio-lubricants is a high viscosity index, or the lubricant’s ability to maintain viscosity under high temperatures.

The reported viscosity index of the estolides produced with oleins was 178 ± five.

This is considered on the lower end. In fact, it was 7% lower than the oleins.

Although estolides produced from

oleins are not as effective on their own, they can act as powerful viscosity modifiers, significantly improving the efficiency of other bio-lubricants.

Although creating an estolide bio-lubricant from oleins was unsuccessful, the experimental studies established fundamental techniques for estolide synthesis. They revealed additional possibilities for estolides in the field of bio-lubricants [14].

More recently, researchers have been focusing on a cleaner approach to estolide synthesis. They’ve been prioritising the biodegradability of the produced estolides and using more environmentally-friendly feedstocks.

Researchers A. Dalai et al developed a novel formulation that transforms canola biodiesel into biodegradable estolides through a mesoporous aluminosilicate-catalyzed reaction [7].

The benefits of facilitating this reaction on the mesoporous aluminosilicate are that the catalyst boasts an extremely high surface area, optimal acid conditions and is compatible with the construction of large organic molecules.

Additionally, the catalyst’s heterogeneous nature makes it easy to separate from the product and reuse it in subsequent formulations.

The resulting estolide was produced with a 96% yield and was tested and characterised as shown in Table 1.

Table 1: Key characteristics of the resulting estolides [7]

Characteristic	
Viscosity index	215
Pour point	-28 ± 2.3 °C
Lubricity or wear scar diameter	106 ± 7.4 µm
Biodegradability	92 ± 4.6% in 28 days

The product was confirmed to be an estolide through H-NMR characterisation.

The viscosity index (VI), an essential indicator of thermal stability, of the product was exceptionally high compared to the previously discussed VI of estolides (around 178 ± five) [15].

This indicates that the produced estolide is a strong candidate as a bio-lubricant.

Looking at the pour point, -28 ± 2.3 °C, this is pretty good for bio lubricants.

The pour point is the lowest temperature at which the estolide

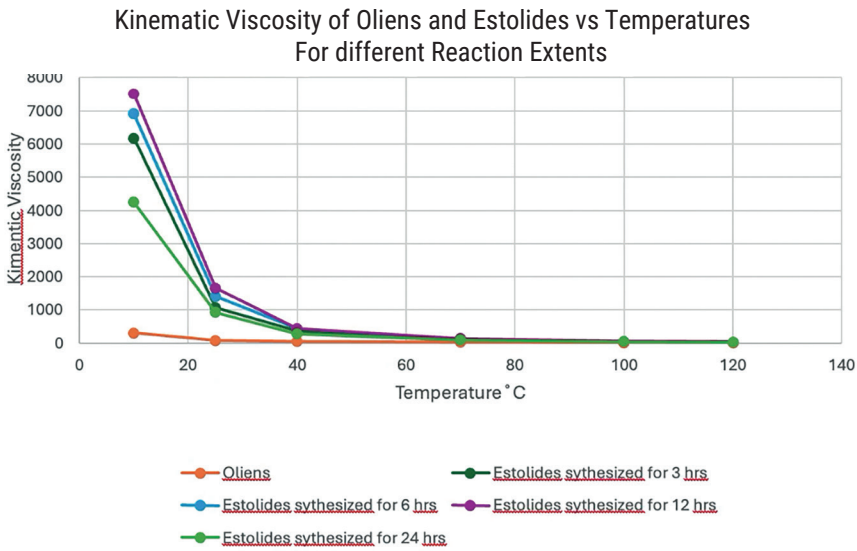


Figure 1: A graph characterising the effect of temperature on the Kinematic Viscosity of different estolide samples and the olein sample [14]

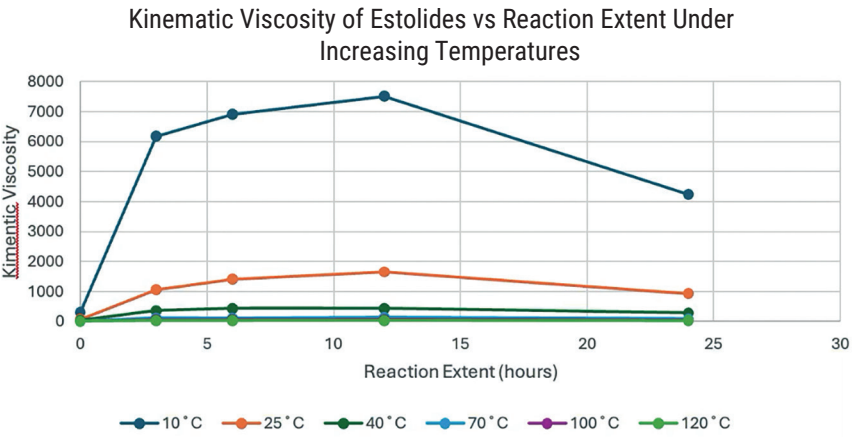


Figure 2: This graph demonstrates the effect of reaction extent on kinetic viscosity under different temperatures

will flow, given the influence of gravity.

The low temperature indicates that this bio-lubricant will still perform at low temperatures.

The lubricity, or the measure of how effective the estolides are at lubrication, is measured with wear scar diameter (WSD). The lower the WSD, the better the lubricant reduces friction.

Compared to pure diesel WSD of 600 μm , the produced estolide offers significantly greater friction reduction as its WSD sits around 106 μm .

The biodegradability of the produced estolides achieved $92 \pm 4.6\%$ aerobic mineralisation within 28 days.

The proposed synthesis strategy is extremely optimal. It offers flexibility in a variety of weather conditions, biodegradability and a clean feedstock option, as well as high yield [7].

Wax esters

Wax esters are organic compounds composed of an ester between a long chain of fatty acid and a long chain of fatty alcohol [10].

In past years, wax esters were obtained from the now-banned whaling methods, specifically from sperm whales.

Currently, methods for securing spermaceti oil remain unsustainable or difficult to implement.

Methods range from cultivating the desert plant Jojoba to chemical processes using petroleum derivatives.

Ultimately, these processes fell out of favour due to high energy costs or difficulty in execution.

However, methods using synthetic biology have been investigated to modify plants to produce wax esters rather than the usual triglycerides.

Researchers K Demski et al focused on genetically engineering the tobacco plant, *Nicotiana benthamiana*, using a mix-and-match approach [10]. Through this trial and error, the researchers were able to formulate a pathway to synthesise wax esters like spermaceti oil to act as a bio-lubricant.

To achieve this, the plants were modified using agrobacterium, following a four-step process.

The first step of this enzymatic pathway is catalyzed by fatty acyl-ACP thioesterases (FAT), which control fatty acid chain length by removing fatty acids of the desired length.

Different FAT enzymes were used to prioritise fatty acid lengths of 12-16 carbons. Then, enzymes from the codling moth and navel orangeworm are added to introduce specific areas of unsaturation into the fatty acid, in the Z or E configuration.

Fatty acyl-CoA reductases (FAR) enzymes are used to convert fatty acids to fatty alcohols.

Finally, the wax ester is assembled with a wax synthase [10].

The desired products were first isolated from the tobacco leaves with thin-layer chromatography, and then the wax esters were decomposed and characterised in gas chromatography.

Ultimately, the results indicated that the produced wax ester resembled that of spermaceti oil.

The 12-16 carbon length chains compositions vary; however, it is more important that we can accurately design wax ester compositions. This was achieved successfully in this experiment.

Although the composition of the target compounds (12:0–16:0 fatty acid and fatty alcohol) moieties was not reported, it was reported that they achieved the highest content of the target compounds.

We are unsure if this process is ready for mass production.

However, this process lays the foundation for genetic engineering targeted in chemical synthesis through synthetic biology.

Interestingly, this method has shown potential for synthesizing insect sex pheromones, with possible uses as environmentally sustainable crop deterrents [10].

Erucic acid



Figure 3: *Brassica carinata* is being grown in a home garden [16]

Erucic acid, a 22-carbon monounsaturated fatty acid, is valued for its varied uses in the biolubricant industry and as a high-quality wax ester feedstock [8].

Similar to the process described earlier, researchers M. Tesfaye et al focused on producing erucic acid and wax esters in a plant setting through genetic modification of plant seeds.

Compared to the previous method, this method is far superior in the industry setting, as the two metabolic pathways introduced into the oilseed crop *B. carinata* become permanent; therefore, the cultivation process following the modification is suitable for industrial mass production [17].

Not only is this developed method suitable for industry demands, but it also produces highly valued erucic acid.

Erucic acid bio-lubricants have desirable properties such as a high viscosity index and thermal stability.

The long carbon chains allow for durable lubrication membranes to form [18]. Feedstocks with high erucic acid content are sought after for their ability to synthesize drop-in biofuels and high-quality wax esters.

Despite erucic acid bio-lubricants being characterised as biodegradable and non-toxic, high erucic acid is undesirable in food-grade oils.

Linkages to heart diseases raise concerns about erucic acid, an anti-nutrient [8]. This may seem alarming, but in the context of erucic acid bio-lubricants, it's beneficial.

A major topic of discussion is the food versus fuel debate. Since erucic acid bio-lubricants are not suitable as edible oils, there is no genuine concern about what the oil's use will be.

Additionally, from an economic perspective, cultivating crops designed for erucic acid production reduces purification costs by half for every 10% increase in erucic acid concentration.

Results from this experiment yielded highs of 52.7%, a 29% increase compared to unmodified or wild *B. carinata* [17].

The modifications not only significantly improve the yield from each acre of land occupied but also reduce costs in the purification and isolation of erucic oil.

Conclusion

The growing field of bio-lubricants offers a cleaner alternative, with many of the advances discussed addressing significant issues, such as performance limitations, sustainability concerns and a lack of technology and infrastructure.

Advances in estolide production have enabled estolides with high yields, high viscosity index and high lubricity. Compared with experiments that established the fundamentals of the estolide output, the biolubrication properties have significantly improved.

Wax ester and erucic acid have both seen advances in synthetic biology. Research focused on the sustainable production of two promising bio-lubricants through genetic engineering of plants.

Overall, advances in technology and knowledge in the field make the future of bio-lubricants ever nearer, with estolides already demonstrating greater thermal resistance than traditional mineral oils [15].

As bio-lubricants grow in popularity, infrastructure will naturally follow. Technology will be developed to optimise production.

Traditional lubricants may have a high bar to meet. But bio-lubricants offer almost limitless potential through the range of possible formulations and organic synthesis methods. Very soon, lithium-based lubricants will be replaced by a much more sustainable, cleaner, and non-toxic alternative of biolubricants.

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Biographies

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Mr Clark Ye is part of a thriving internship program at Koehler Instrument Company in Holtsville.



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