PAPER

Sustainable base oils from non-edible fats: performance of esterified, epoxidized, and hydroxylated brown grease

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Brown grease (BG), a high free-fatty-acid rendering coproduct, was upgraded to lubricant base stocks *via* one of four single-step functionalization methods: ethyl esterification, methyl esterification, epoxidation, or hydroxylation to give **FAEEc/b**, **FAMEc/b**, **Epoxy BGc/b**, or **Hydro BGc/b**, respectively (from crude, c, or bleached, b, BG). Each modification was carried out on crude and degummed/bleached brown grease, and the ten materials were evaluated by thermogravimetric analysis ($T_{d,5\%}$), dynamic (μ) and kinematic (ν) viscosities at 40 and 100 °C, density, viscosity index (VI), and pour point, then mapped to ISO VG grades. Clear structure–property trends emerge. Esterification lowers ν_{40} and improves temperature-thinning (**FAEEb**: $\nu_{40} = 9.33$ mm^2 s^{-1}, VI = 199, pour point = 0 °C; ISO VG 10). Hydroxylation raises ν_{40} into mid-grade ranges (**Hydro BGc**: $\nu_{40} = 98.4$ mm^2 s^{-1}, $\nu_{100} = 12.5$ mm^2 s^{-1}, VI = 121; ISO VG 100) and delivers the highest thermal onset ($T_{d,5\%} = 234$ °C), but with poor cold-flow (pour point = 28 °C). Epoxidation affords very high viscosities ($\nu_{40} = 1.5$–1.7×10^3 mm^2 s^{-1}, VI = 30–40), best suited as thickeners rather than neat base stocks. Parent greases fall near ISO VG 32 ($\nu_{40} = 34$–38 mm^2 s^{-1}) with pour points of 28–31 °C. These proof-of-concept results delineate application-relevant niches (high-VI light esters; VG 100 hydroxy-oils) and demonstrate a route to valorise non-edible fats into bio-derived lubricants.

Introduction

Lubricants are ubiquitous in manufacturing, power generation, and agricultural applications, and are essential to attenuate wear, manage heat stress, and protect surfaces.[1] Most base stocks for commercial lubricants are petroleum-derived, contributing to the fossil carbon footprint and persistent environmental burdens from their release into the air and water.[2,3] Policy and market forces thus call for more renewable surrogates.[4] Recent reviews delineate significant progress in greener lubricant solutions and provide a common framework for evaluating candidate base oils based on kinematic viscosity at 40 and 100 °C (ν_{40} and ν_{100}), viscosity index (VI) and pour point.[5–7]

First-generation bio-based oils have most often been prepared from edible plant oils (*e.g.*, soybean, canola, palm), intensifying competition with food systems and land use and raising costs.[8–11] The food security challenges facing humanity, however, call for a departure from using edible feedstocks for non-edible goods.

To avoid "food *versus* materials" conflicts, recent work has prioritized non-edible, residual lipid streams that cannot be used for human or animal consumption. One promising triglyceride source is brown grease, also called grease trap

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waste. Brown grease is recovered from food service and drainage systems and typically has a high free fatty acid (FFA) content and may contain tri/di/monoglycerides, water, solids, phospholipids, pigments, and trace metals, making it unsuitable for nutritional products for human or animal markets.[12–14] Brown grease thus often has low or negative net value due to disposal costs. Recent work has revealed that brown grease can replace plant oils in bio-derived structural materials like plastic cements[15–19] and polyurethanes.[8–11] Brown grease has also been explored as a feedstock for bio-derived fuels, but its chemical characteristics complicate its use in fuel applications.[20–25]

The biolubricants literature provides strong precedent for triglyceride-derived base oils,[26] though raw triacylglycerides seldom suffice without initial chemical modification. Synthetic esters, notably pentaerythritol and trimethylolpropane polyol esters, have found commercial application in aviation, refrigeration, and hydraulic systems as they afford relevant VIs, flow, film-forming capacity, and thermophysical properties.[27,28] Estolides, fatty acid oligomers produced by self- or cross-condensation, have also been commercialized in multiple market segments.[29] Although unmodified vegetable oils display inherent drawbacks, these shortcomings are mitigated by chemical modification, notably transesterification,[30] epoxidation,[5] and hydroxylation.[31] As a more affordable triglyceride source, brown grease similarly presents an opportunity for potential chemical upgrading to lubricant base stocks.

In the current work, we evaluate the viability of brown grease as a base oil precursor towards eventual use of brown grease derivatives in lubricant applications. Chemical modification of brown grease *via* esterification to give fatty acid ethyl esters (**FAEEx**) or fatty acid methyl esters (**FAMEx**), epoxidation to give **Epoxy BGx**, and hydroxylation to give **Hydro BGx** (x = c or b for products from crude brown grease or bleached brown grease, respectively). Ten brown grease-based materials were evaluated for thermal stability, dynamic (μ) and kinematic (ν) viscosities at 40 and 100 °C, density, viscosity indices (VI), and pour points, with mapping to ISO VG grades and benchmarking against commercial standard materials and other bio-derived lubricants and base oils.

Results and discussion

Pretreatment and chemical modification of Brown grease

Crude brown grease (**BGc**) obtained from a commercial rendering plant was initially characterized by a combination of ^{1}H NMR spectroscopy and UV-vis colorimetric analysis using the CDR FoodLab. Using 2,3,4,5,6-pentafluorobenzaldehyde (PFBA) as an internal standard, ^{1}H NMR spectroscopy revealed an olefin content of 11 mmol of C=C per gram of oil, corresponding to an iodine value of 280. UV-vis colorimetric analysis using the CDR FoodLab revealed a free fatty acid content of 59% and a peroxide value of 10 mmol O_2 per kg oil (Table 1 and SI Fig. S1).

BGc was conditioned by an acid hydration degumming protocol adapted from procedures reported to condition other bio-derived oils.[32] Briefly, the oil was stirred at an elevated temperature to homogeneity and phosphoric acid was added, followed by water. Gravity separation afforded a lower aqueous/phospholipid phase that was discarded. The upper oil phase was clarified by vacuum filtration over diatomaceous earth (Celite®) filtration aid, and residual acid was removed by recursive water washes. The degummed material exhibited a free fatty acid content of 60%, similar to that of **BGc**.

Degummed brown grease was subjected to an additional bleaching step designed to remove chromophores and trace impurities that could potentially complicate subsequent oxidations and influence lubricant properties (*vide infra*).[33] For the bleaching step, degummed brown grease was heated with activated bleaching clay and activated carbon as primary scavengers, and Celite® was added as a filtration aid. The spent solids were removed by vacuum filtration, and the bleached brown grease (**BGb**) was collected and characterized by ^{1}H NMR spectroscopy and UV-vis colorimetric analysis. These tests revealed an olefin content of 12 mmol olefin per g oil (corresponding to an iodine value of 300), a free fatty acid content of 63% and a peroxide value of 8.4 mmol O_2 per kg oil (Table 1, spectra provided in SI Fig. S2).

Chemical modification: esterification

Brown grease contains free fatty acids (FFAs) and triacylglycerols in proportions that vary by source and storage conditions. Esterification by reaction with methanol to give fatty acid methyl esters (FAMEs, Scheme 1A) or with ethanol to give fatty acid ethyl esters (FAEEs, Scheme 1A) converts all fatty acid chains to esters, neutralizing any effects of this FFA content variability. Esterification of crude brown grease and bleached brown grease by modified literature protocols[21] gave **FAMEx**/**FAEEx** (x = c or b for products from **BGc** or **BGb**, respectively). Proton NMR spectrometric analysis of the resulting mixtures was used to assess the extent of conversion. The FAME mixture showed a singlet at 3.66 ppm attributable to the installed –OCH$_3$ group. Comparing integration of the methoxy resonance against the terminal methyl resonances, shown as a broad singlet near 0.875 ppm, confirmed desired transformation to **FAMEc** and **FAMEb** in moderate yield (51–70%, spectra provided in SI Fig. S3–6).

Diagnostic resonances in the FAEE mixture characteristic of –OCH$_2$– at ≈ 4.13 ppm and CH$_3$CH$_2$– triplet at ≈ 2.28 ppm were integrated with respect to terminal methyl resonances, to confirm conversion from crude and bleached brown grease, respectively, giving **FAEEc** and **FAEEb** in good yield (60–89%, spectra provided in SI Fig. S7–10). Table 1 summarizes additional metrics, including the expected decrease in free fatty acid content to <10%, and olefin content ranging from 6.4 to

Table 1 Compositional metrics for brown grease (crude, bleached) and derived products: free fatty acid content, peroxide value, iodine value, and olefin content

| Sample name	Free fatty acid content[a] (%)	Peroxide value[a] (mmol O_2 per kg oil)	Iodine value[b] (g I$_2$/1 per 00 g oil)	Olefin content[c] (mmol olefin per g oil)
Brown grease (crude)	59	10	280	11
Brown grease (bleached)	63	8.4	300	12
FAEEc	10	58	320	13
FAEEb	6.0	24	170	6.8
FAMEc	4.0	45	270	11
FAMEb	2.0	27	160	6.4
Hydro BGc	40	8.5	110	4.2
Hydro BGb	41	13	56	2.2
Epoxy BGc	68	9.2	10	0.41
Epoxy BGb	56	9.6	15	0.58

[a] Free fatty acid content and peroxide value measured on FoodLab. [b] Iodine value (IV) calculated as IV = 25.38 × (mmol C=C per g oil). [c] Olefin content (mmol C=C per g oil) from ^{1}H NMR using PFBA internal standard.
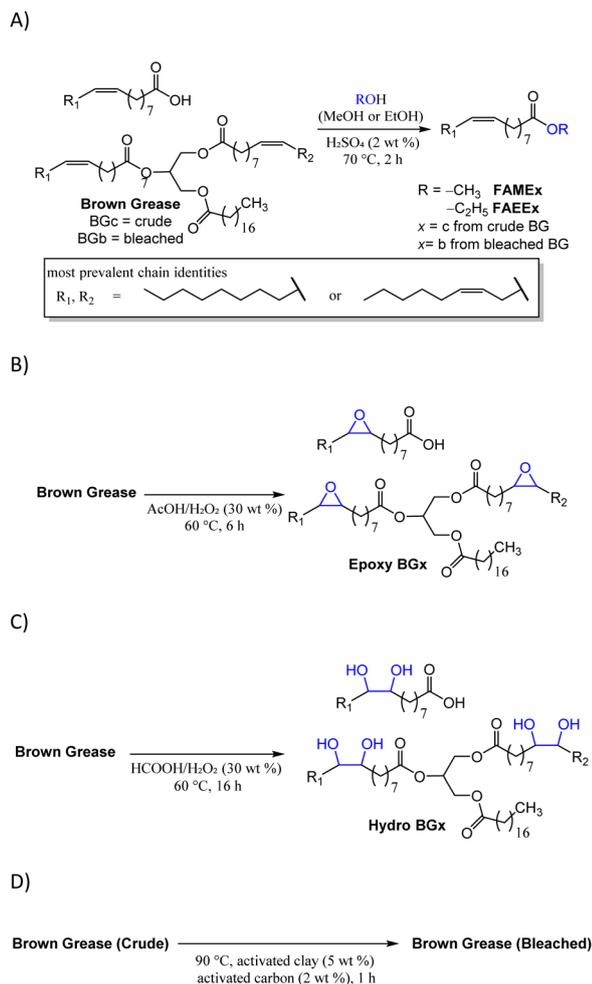
Scheme 1 Chemical modification of brown grease by (A) esterification, (B) epoxidation, and (C) hydroxylation. x = c denotes materials made from crude brown grease, while x = b denotes materials made from bleached brown grease. (D) Procedure for bleaching brown grease.

13 mmol olefin per g oil. It is noted that there is a significant increase in peroxide values for the esterified materials, ranging from 24–58 mmol O_2 per kg oil, attributable to the carryover from the starting material and exposure to ambient oxygen during reactions. Esterified materials were isolated as dark brown liquids (Fig. 1).
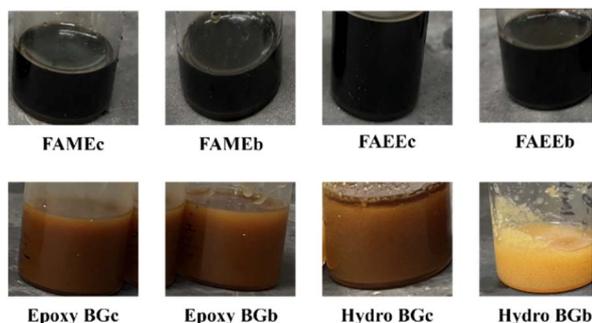
Fig. 1 Pictures of the chemically modified brown grease oil.

Chemical modification: epoxidation

Unsaturated sites in samples of crude and bleached brown grease were converted to epoxides *via* a Prilezhaev epoxidation using a peracetic acid generated *in situ* from acetic acid and hydrogen peroxide (Scheme 1B).[34] This process gave **Epoxy BGx** (x = c or b for products from **BGc** or **BGb**, respectively). For the crude brown grease (**BGc**), incomplete conversion was initially observed under initial epoxidation conditions, with residual olefinic resonances persisting in the ^{1}H NMR spectrum. An incomplete reaction was visually evident as well by observation of a two-phase mixture during the reaction (SI Fig. S11). To achieve comparable levels of conversion to those obtained with the bleached feed (**BGb**), it was necessary to employ higher equivalents of both hydrogen peroxide and acetic acid, confirming that impurities in the crude brown grease consume some oxidizing equivalents during reaction.[5–7]

Proton NMR spectrometric analysis gave evidence for consumption of olefins by near complete disappearance of olefinic proton resonances at 5.34 ppm with proportional growth of resonances near 1.43 ppm attributable to epoxide methine units (SI Fig. S12–15). FTIR spectral analysis provided further evidence in the form of the emergence of epoxide ring bands at ≈ 1230 cm^{-1}, concurrent with disappearance of the olefinic =C–H stretch at ≈ 3005 cm^{-1} (SI Fig. S16–19 and 36). UV-vis colorimetric analysis (Table 1) showed no significant change in free fatty acid content, ranging from 56–68%, and peroxide values ranging from 9.2–9.6 mmol O_2 per kg oil. The epoxidized oils derived from crude and bleached brown grease were isolated as thick brown waxy substances (Fig. 1) in 80–81% yield.

Chemical modification: hydroxylation

Hydroxylated derivatives **Hydro BGx** (x = c or b for products from **BGc** or **BGb**, respectively) were prepared by initial epoxidation by performic acid generated *in situ* from formic acid and hydrogen peroxide, followed by water-mediated ring-opening and protonation in a one-pot process (Scheme 1C). These materials were analysed by Infrared spectroscopy (SI Fig. S20–21 and 36), proton NMR spectrometry (SI Fig. S22–25) and UV-vis colorimetric analysis, providing evidence for successful hydroxylation. In the ^{1}H NMR spectra, the olefinic resonance at ≈ 5.34 ppm is reduced greatly, and a new signal at 1.49 ppm appears, consistent with the expected formation of –CH(OH)– units resulting from epoxide ring-opening. These spectroscopic changes track with the drop in olefin content from 11 to 4 mmol olefin per g oil and 12 to 1.4 mmol g^{-1} oil for the **Hydro BGc** and **Hydro BGb**, respectively. Hydroxylated oils prepared from crude and bleached brown grease were isolated as light brown liquids (Fig. 1) in $\approx 80\%$ yield. Integration of chain-terminating methyl *versus* internal methylene resonances in the proton NMR spectra support the integrity of the chains initially present in the BGc and BGb samples following chemical modification steps (SI Fig. S1–10 and S22–25).

Thermal stability

Thermogravimetric analysis (TGA) was used to assess the onset of volatilization/thermal decomposition for the crude brown grease, bleached brown grease, and the eight brown-grease-

derived modified oils (Table 2 and SI Fig. S26–36). For each sample, the temperature at five percent mass loss under nitrogen flow ($T_{d,5\%}$) was taken as a comparative indicator of thermal stability. Hydroxylated materials exhibited the highest $T_{d,5\%}$, with **Hydro BGc** reaching 234 °C. Epoxidized derivatives fell in an intermediate range, with **Epoxy BGc** at 211 °C and **Epoxy BGb** at 229 °C. The parent brown grease samples were near 209 °C irrespective of bleaching. Fatty acid alkyl esters showed the lowest $T_{d,5\%}$, with **FAMEb** at 183 °C, **FAMEc** at 190 °C, **FAEEb** at 196 °C, and **FAEEc** at 200 °C.

These trends are consistent with previous observations on the thermal stability of analogously modified triglycerides such as plant oils, and are consistent with the expected structural modifications introduced by each route.[35–37] Hydroxylation can provide stabilization *via* hydrogen bonding and elimination of unsaturated sites. Epoxidation likewise removes thermally susceptible allylic and unsaturated sites while stiffening the hydrocarbon backbone. In contrast, esterification reduces polarity, but does not change the unsaturation content, yielding materials with relatively lower $T_{d,5\%}$ values within the series, but that are nevertheless compatible with moderate-temperature lubricant service applications. For the epoxidized oils, bleaching prior to oxidation increased $T_{d,5\%}$ (from 211 to 229 °C), consistent with removal of colour bodies and trace metals that catalyse epoxide ring opening and downstream thermal decomposition. In the hydroxylated materials, however, the bleached sample showed a lower $T_{d,5\%}$ than its crude counterpart (234 *versus* 215 °C), indicating that the increased polarity and hydrogen bonding of the diol-rich product dominate over any stabilizing effect of impurity removal. In the esterified materials, bleaching likewise produced a small downward shift in $T_{d,5\%}$ (*e.g.*, FAEE from 200 to 196 °C and for FAME 190 to 183 °C), which may reflect the combined effects of slightly lower average molecular weight and reduced polar impurity content that otherwise contribute to intermolecular cohesion in the crude feed. Finally, the negligible difference between crude and bleached brown grease underscores that in the absence of further functionalization, the bulk cohesion of the triglyceride/FFA mixture is only weakly modulated by the trace impurities removed during bleaching.

Comparison of the thermal properties of the brown grease-derived materials (Table 2) against conventional base-oil families (Table 3) reveals that **Hydro BGc** at 234 °C approaches the decomposition-onset range of Group III mineral oils[38] and some synthetic esters,[39] whereas the epoxidized and parent greases occupy a mid-tier band. The esterified materials, particularly **FAMEb**, display $T_{d,5\%}$ values characteristic of light ester stocks and biodiesel-type fluids.[40]

Viscosity grade, rheology, and viscosity index

Kinematic viscosity at 40 (ν_{40}) and 100 °C (ν_{100}) and the derived viscosity indices (VI) were measured for the ten brown-grease-derived base stocks to assess potential temperature-thinning behaviour and to assign standard ISO viscosity grades.[41] The data span more than two orders of magnitude in ν_{40} and ν_{100} (Table 2), from light ester fluids in the ISO VG 10 class to highly viscous epoxidized oils outside the ISO scale, providing a useful measure of structure–property relationships resulting from four chemistries and the crude/bleached feed treatments.

The esterified materials have the lowest kinematic viscosity values of the series. **FAEEx** and **FAMEx** give $\nu_{40} \approx$ 9–12 mm^2 s^{-1} and $\nu_{100} \approx$ 2.5–3.0 mm^2 s^{-1}, corresponding to ISO VG 10. Bleaching BG has a notably positive effect on temperature-thinning for **FAEEx**. The VI rises from 93.6 for **FAEEc** to 199 for **FAEEb** at essentially the same ν_{40} (11.7 and 9.33 mm^2 s^{-1}) and ν_{100} (2.90 and 2.98 mm^2 s^{-1}). This behaviour is consistent with removal of impurities that contribute to non-ideal thinning and with the shift from a mixed FFA/mixed glyceride matrix to a more uniform esterified fluid. By contrast, FAME shows only modest VI values ($\approx$ 83–100) and similar ν_{40} (11.7 *versus* 9.59 mm^2 s^{-1}) and ν_{100} (2.93 *versus* 2.52 mm^2 s^{-1}) values regardless of BG pre-treatment. **FAMEx** metrics indicate a light stock oil that remains more temperature-sensitive than **FAEEx** at comparable viscosity. In practical terms, **FAEEx** and **FAMEx** materials resemble spindle-oil-class fluids.[42–44] **FAEEb** approaches synthetic-ester-like VI at very low viscosity, whereas **FAMEx** approximates a conventional light ester in its rheological profile.[45]

Table 2 Thermal and rheological properties of brown grease and derived oils. Thermal properties reported as temperature at 5% weight loss ($T_{d,5\%}$)

Sample name	$T_{d,5\%}$ (°C)	Dynamic viscosity (mPa s)		Density (g mL^{-1})		Kinematic viscosity (mm^2 s^{-1})		Viscosity index (VI)	Pour point (°C)
		40 °C (μ_{40})	100 °C (μ_{100})	40 °C (ρ_{40})	100 °C (ρ_{100})	40 °C (ν_{40})	100 °C (ν_{100})		
Brown grease (crude)	209	35	6.68	0.92	0.89	38	7.51	170	28
Brown grease (bleached)	209	31.3	5.05	0.92	0.87	34	5.8	112	31
FAEEc	200	10.4	2.52	0.888	0.868	11.7	2.9	93.6	3
FAEEb	196	8.35	2.52	0.895	0.845	9.33	2.98	199	0
FAMEc	190	10.5	2.52	0.898	0.861	11.7	2.93	99.8	3
FAMEb	183	8.45	2.15	0.881	0.854	9.59	2.52	83.1	3
Hydro BGc	234	95.4	11.5	0.97	0.92	98.4	12.5	121	28
Hydro BGb	215	95.5	7.55	0.962	0.921	99.3	8.2	12.4	28
Epoxy BGc	211	1590	40.5	0.92	0.903	1730	44.9	30.3	41
Epoxy BGb	229	1440	39.3	0.946	0.901	1520	43.6	39.6	41

Table 3 Commercial/literature base-oil benchmarks: thermal and rheological metrics. Thermal properties reported as temperature at 5% weight loss. PPD = pour point depressant

Commercial/literature base oil	$T_{d,5\%}$ (°C)	v_{40} (mm² s⁻¹)	v_{100} (mm² s⁻¹)	VI	Pour point (°C)	ISO VG (typical)	Notes
Mineral oil (Group I)	≈ 240	32–150	4–15	90–100	−9 to −18	32–150	Baseline mineral; modest VI, moderate pour point; needs VIIs/PPDs
Mineral oil (Group III)	250–260	32–68	4–10	120–140	−18 to −27	32–68	High VI mineral; good low-T; widely used
PAO (poly-α-olefin)	260–280	32–100	5–15	130–160	−45 to −60	32–100	Synthetic standard; excellent cold flow and VI.
Synthetic esters (TMP/diesters)	240–270	8–68	3–12	140–200	−40 to −60	10–68	Aviation/industrial base stocks; high VI and excellent pour points
Estolides (oleic/iso-oleic, incl. 2-EH esters)	Varies	Varies	—	141–170	−41 to −60	Varies	Bio-derived base oils with outstanding cold flow; tunable viscosity
Polyol esters (TMP trioleate)	Varies	45–55	9–10	180–183	≤−30	≈ 46	Common bio-ester base fluid; good VI and pour points
High-oleic sunflower oil (HOSO)	Varies	30–40	—	≥220	Varies	32–46	Raw oil with very high VI; usually needs PPD and antioxidants

The brown grease precursors themselves are in lower ISO industrial grades. The crude grease exhibits $v_{40} = 38.0$ mm² s⁻¹ and $v_{100} = 7.51$ mm² s⁻¹, correlating to ISO VG 32, with a relatively high VI of 170, reflecting a mixed FFA/glyceride composition that thins less than a simple low-molecular ester at elevated temperature. After bleaching, v_{40} decreases to 34.0 mm² s⁻¹ and v_{100} to 5.80 mm² s⁻¹, and the VI drops to 112. This reduction indicates that components removed in the bleaching step contributed to high-temperature viscosity retention in the crude feed; their removal yields a cleaner but more temperature-sensitive fluid at similar v_{40}.

Hydroxylation produces materials better approaching useful hydraulic/gear-oil viscosities. **Hydro BGc** shows $v_{40} = 98.4$ mm² s⁻¹ and $v_{100} = 12.5$ mm² s⁻¹, meeting ISO VG 100, with a moderate VI of 121. Increased hydrogen-bonding is generally credited with the viscosity elevation and the improved retention of viscosity at 100 °C relative to light esters. Notably, bleaching prior to hydroxylation yields $v_{40} ≈ 99$ mm² s⁻¹ but reduces v_{100} to 8.20 mm² s⁻¹, collapsing the VI to 12.4. This dramatic change suggests that some as-yet unidentified impurities in the crude feed contribute to networked interactions that help sustain high-temperature viscosity in the hydroxylated product.

Epoxidation produces the highest-viscosity and lowest VI oils in the set. **Epoxy BGc** and **Epoxy BGb** display v_{40} of 1730 and 1520 mm² s⁻¹, respectively, with v_{100} near 44–45 mm² s⁻¹ and VI in the 30–40 range. These values lie far outside the ISO VG scale for neat base-oil application, suggesting these materials may be more suitable as thickeners. The low VI reflects strong temperature-dependent relaxation, with high zero-shear viscosity dominated by backbone stiffening. Although bleaching prior to epoxidation does reduce v_{40} modestly and raise T_d, it does not alter the qualitative rheological classification of **Epoxy BGb** compared to that of **Epoxy BGc**.

Low-temperature properties (pour point)

Pour point measurements provide a first screening of cold-flow behaviour and were used in the current study as a useful comparative index across brown grease-derived materials. The brown-grease-derived materials exhibit pour points ranging from 0–41 °C. The esterified materials are most promising for conventional applications in this regard, with FAEE showing the lowest values in the series, improving from 3 °C (crude feed) to 0 °C upon bleaching, and FAME remains at 3 °C irrespective of bleaching status. By contrast, both the parent greases and the more polar derivatives exhibit poor cold flow. The crude and bleached brown greases span 28 to 31 °C, hydroxylated oils remain at 28 °C regardless of pretreatment, and epoxidized oils are highest at 41 °C for both crude- and bleached-feed variants. Comparison of pour point data with DSC (differential scanning calorimetry) analysis revealed that esterified oils exhibit crystallization onsets near −5 °C, consistent with measured pour points (0 °C ± 3 °C) (SI Fig. S37 and 38). The hydroxylated oils exhibit a crystallization onset near 1 °C, while the epoxidized samples show distinct crystallization around 18 °C (SI Fig. S39 and 40), highlighting differences between the measured pour points. These differences are likely attributable to high polarity and intermolecular hydrogen bonding, rather than crystallization transitions. These trends are consistent with the expected role of polarity and microstructure in low-temperature behaviour, as observed for similarly modified plant oil-derived lubricants.

Relative to commercial benchmarks, even the esterified materials exhibit pour points substantially higher than those typical of PAO (≈ −45 to −60 °C) and synthetic polyol/diesters (≈ −40 to −60 °C), and higher than reported values for many estolides (≈ −41 to −60 °C). Given that automotive and many other industrial fluids commonly require pour points ≤−30 °C, pour point depressants could be considered for future work aimed at improving this aspect of the materials.

Benchmarking and application mapping

The BG-derived fluids' most application-relevant neat base stocks are **FAEEb** (ISO VG 10 with VI = 199) and **Hydro BGc** (ISO VG 100 with VI = 121). Against commercial and plant-oil benchmarks (Tables 3 and 4),[5–7,41] **FAEEb** approaches

Table 4 ISO viscosity-grade assignments and application notes for brown-grease-derived oils

Sample	ISO VG (by ν_{40})	Notes
Brown grease (crude)	32	High VI for a raw feed, but very high pour point; unsuitable neat
Brown grease (bleached)	32	Lower ν and VI vs. crude; poor cold flow
FAEEc	10	Light ester; VI modest, cold flow reasonable
FAEEb	10	Top candidate: very high VI ($\approx$ synthetic esters); cold flow $\approx$ 0 °C; light viscosity
FAMEc	10	Biodiesel-like methyl esters; light & modest VI.
FAMEb	10	Lower VI and ν; light stock with limited temperature robustness
Hydro BGc	100	ISO VG 100 with decent VI & high Td
Hydro BGb	100	VI collapses after bleaching; unusable neat
Epoxy BGc	>1500 (classify as thickener)	Extremely viscous, poor VI; use as viscosity modifier only
Epoxy BGb	>1500 (classify as thickener)	Similar to crude epoxy; too viscous for base-oil use

synthetic-ester-like VI at light viscosity (albeit in warm/controlled environments due to high pour point), whereas **Hydro BGc** provides a VG 100 anchor with adequate thermal properties but limited cold-flow. Epoxy BG is positioned as a viscosity modifier or thickener (ν_{40} > 1500, VI = 30.3–39.6). **BGc/BGb** cluster near ISO VG 32 but retain high pour points (28 and 31 °C).

Preliminary tribological tests were performed using a standard four-ball wear configuration (ASTM D4172) for two representative candidates, **FAEEc** and **Hydro BGc**, at 75 °C and 1200 rpm. The measured average wear scar diameters were 0.80 mm for **FAEEc** and 0.70 mm for **Hydro BGc** (four-ball wear graph with coefficient of friction shown in SI Fig. S41 and 42). These results are slightly higher compared to other bio-based lubricants, such as cottonseed oil (CSO) and waste cooking oil (WCO), whose average wear scar diameters were 0.60 and 0.55 mm, respectively.[46] Polyalphaolefin (PAO), a common synthetic base oil, had an average wear scar diameter of 0.61 mm without any additives.[46] For **FAEEc**, the coefficient of friction initially rose from 0.06 to 0.08 over the first 30 minutes of testing, then remained nearly constant around 0.075 for the remaining 90 minutes of testing (SI Fig. S41). For **Hydro BGc**, the coefficient of friction initially dropped from 0.08 to 0.05 over the first 30 minutes, then remained at 0.05 ± 0.006 for the remaining 90 minutes of testing (SI Fig. S41). Coefficients of friction trends for these BG-derived materials are again similar to those of the aforementioned CSO, WCO, and PAO examples. Further research will investigate methods to enhance wear performance through additional chemical modifications and the incorporation of additives.

Conclusions

Brown grease (BG), a non-edible, high-FFA rendering coproduct, was converted by one-pot functionalization steps by either ethyl/methyl esterification, epoxidation, or hydroxylation, to give ten base-oil candidates from crude and bleached BG feeds. The ten candidates were evaluated for ν_{40}/ν_{100}, μ_{40}/μ_{100}, ρ, VI, pour point, and $T_{d,5\%}$. Some general structure–property relationships emerge.

Esterification lowers viscosity and improves temperature-thinning, with **FAEEb** having ν_{40} = 9.33 mm² s⁻¹ (ISO VG 10),

VI = 199, pour point = 0 °C, i.e., a high-VI light base stock. Hydroxylation raises viscosity into mid-grades. **Hydro BGc** had ν_{40} = 98.4 mm² s⁻¹, ν_{100} = 12.5 mm² s⁻¹, VI = 121, placing it in ISO VG 100, and hydroxylated products had the highest thermal stability ($T_{d,5\%}$ = 234 °C), but at the expense of cold-flow (pour point = 28 °C).

Epoxidation produces thickener-class liquids (ν_{40} = 1.5–1.7 × 10³ mm² s⁻¹, VI = 30–40, pour point = 41 °C). The parent greases cluster near ISO VG 32 (ν_{40} = 34–38 mm² s⁻¹) with pour points (28 to 31 °C) above the typical useful range.

Pretreatment exerts chemistry-dependent effects: bleaching improves VI for FAEE at essentially constant viscosity grade, moderately increases $T_{d,5\%}$ for epoxides, and collapses VI for hydroxylated products despite similar ν_{40}.

Benchmarking against commercial and plant-oil-derived base stocks suggests two immediate potential application niches: (i) high-VI light esters for warm/controlled environments, and (ii) VG 100 hydroxy-oils where viscosity class and thermal headroom are paramount but cold-flow is non-critical. Simple viscosity-blending calculations indicate that FAEE (bleached)/Hydro BG (crude) pairs can be tuned to common ISO grades (VG 15–68) with VI superior to Hydro alone, though lower temperature operation will still require cold-flow solutions.

Overall, the present study establishes a comprehensive baseline for brown-grease-derived base stocks accessed via four chemical modification processes using two feed brown grease products. Future work will focus on improving lubricant metrics and subjecting more promising candidates to more complete testing involving dynamic low-temperature rheology (MRV/CCS), oxidative and shear stability, tribology, and additive/blend optimization to translate these base stocks into formulated lubricants.

Experimental

Materials and methods

Reagents were used as received from Millipore Sigma, Acros Organics, and Thermo Fisher Scientific. Suppliers for brown grease were omitted to avoid endorsement or bias. Proton NMR spectra were obtained on a Bruker NEO-300 operating at 300 MHz at 20–23 °C. Chemical shifts (δ) are reported in parts per million (ppm). Spectra data were processed with SpinWorks

4.2.11 software and referenced to residual solvent peak (^{1}H, CDCl$_3$, 7.26 ppm). Multiplicity is reported as s, singlet; d, doublet; and m, multiplet. Fourier transform infrared spectra were collected on Shimadzu IR Affinity-1S with an ATR attachment operating over 400–4000 cm^{-1} at 20–23 °C. Thermogravimetric analysis (TGA) was performed, and data were collected on a Mettler Toledo TGA 2 STARe System over the range of 25–800 °C, with a heating rate of 10 °C min^{-1} under a flow of N$_2$ at a rate of 20 mL min^{-1}. Free fatty acid content (%) and peroxide value (mmol O$_2$ per kg oil) obtained using CDR FoodLab per manufacturer protocol. Dynamic viscosity (μ) at 40 and 100 °C was measured on an Anton Paar MCR 302e rheometer using measuring cone CP50-0.5; angle 0.5° geometry at a constant shear rate. Kinematic viscosity was calculated as $\nu = \mu/\rho$, and density (ρ) was calculated by volume displacement at 40 and 100 °C. Viscosity Index (VI) calculated from ν_{40} and ν_{100} according to ASTM D2270.[47] Pour point was measured per ASTM D97.[48] Differential scanning calorimetry (DSC) data were acquired (Mettler Toledo DSC 3 STARe System) over a temperature range from −60 to 140 °C with a heating rate of 10 °C min^{-1} under a flow of N$_2$ (200 mL min^{-1}). Each DSC measurement was carried out over three heat-cool cycles.

Degumming (acid hydration) of crude brown grease

To a 1 L round-bottom flask, 208.7 g of **BGc** was added, heated to 80 °C using an oil bath, and stirred at 400 rpm. To the **BGc**, phosphoric acid (0.2 wt% relative to oil) was added, mixed for 30 minutes, then 300 mL of DI water was added and stirred for an additional 60 min at 80 °C. After 1 hour, 12 g of NaCl was added to reduce emulsification, and the reaction mixture was allowed to phase separate at 80 °C. The lower aqueous layer was removed, and the oil was collected for subsequent bleaching step (127.3 g, 61%). Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 5.34 (m, **H**–C=C–, 2H); 2.35 (m, –**CH$_2$**–CO, 2H); 2.01 (m, **CH$_2$**–C=C–**CH$_2$**, 3H); 1.63 (m, –**CH$_2$**–CCOO, 2H); 1.25 (m, –**CH$_2$**–, 20H); 0.85 (m, –**CH$_3$**, 3H).

Bleaching of brown grease

To a 250 mL round-bottom flask, the degummed BG was added, heated to 90 °C using an oil bath, and agitated at 500 rpm. To the degummed BG, activated bleaching clay (5 wt%), activated carbon (2 wt%), and diatomaceous earth (5 wt%) were added. The mixture was allowed to stir at 90 °C under vacuum for 1 hour, after which the reaction mixture was filtered to afford **BGb** (104.9 g, 82%). Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 5.34 (m, **H**–C=C–, 2H); 2.35 (m, –**CH$_2$**–CO, 2H); 2.01 (m, **CH$_2$**–C=C–**CH$_2$**, 3H); 1.63 (m, –**CH$_2$**–CCOO, 2H); 1.25 (m, –**CH$_2$**–, 20H); 0.85 (m, –**CH$_3$**, 3H).

General esterification to FAME and FAEE (FAMEx/FAEEx)

A 100 mL round-bottom flask equipped with a magnetic stir bar was placed in an oil bath on top of a magnetic stir plate. To the round-bottom flask, **BGx** (10 g), methanol or ethanol (90 mmol), and sulfuric acid (2 wt%) were added sequentially. The reaction flask was equipped with a condenser and heated to reflux at 70 °C for 2 hours. Upon completion of the reaction, the mixture was

dissolved in 30 mL of diethyl ether, washed twice with 30 mL aqueous solution of 5 wt% sodium bicarbonate, followed by two washes with 30 mL of DI water. The organic phase was then collected, dried under magnesium sulfate for 15 minutes, vacuum-filtered to remove salts, and concentrated under reduced pressure to afford a dark brown oil.

Characterization of FAMEc

70% yield. Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 5.34 (m, **H**–C=C–, 2H); 3.66 (s, O–**CH$_3$**, 2H); 2.30 (m, –**CH$_2$**–CO, 2H); 2.01 (m, **CH$_2$**–C=C–**CH$_2$**, 3H); 1.61 (m, –**CH$_2$**–CCOO, 2H); 1.25 (m, –**CH$_2$**–, 19H); 0.85 (m, –**CH$_3$**, 3H).

Characterization of FAMEb

51% yield. Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 5.34 (m, **H**–C=C–, 2H); 3.66 (s, O–**CH$_3$**, 2H); 2.30 (m, –**CH$_2$**–CO, 2H); 2.01 (m, **CH$_2$**–C=C–**CH$_2$**, 3H); 1.61 (m, –**CH$_2$**–CCOO, 2H); 1.25 (m, –**CH$_2$**–, 20H); 0.85 (m, –**CH$_3$**, 3H).

Characterization of FAEEc

60% yield. Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 5.34 (m, **H**–C=C–, 2H); 4.13 (s, O–**CH$_2$CH$_3$**, 1H); 2.28 (m, –**CH$_2$**–CO, 2H); 2.01 (m, **CH$_2$**–C=C–**CH$_2$**, 3H); 1.61 (m, –**CH$_2$**–CCOO, 2H); 1.25 (m, –**CH$_2$**–, 21H); 0.85 (m, –**CH$_3$**, 3H).

Characterization of FAEEb

89% yield. Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 5.34 (m, **H**–C=C–, 2H); 4.13 (s, O–**CH$_2$CH$_3$**, 1H); 2.28 (m, –**CH$_2$**–CO, 2H); 2.01 (m, **CH$_2$**–C=C–**CH$_2$**, 3H); 1.61 (m, –**CH$_2$**–CCOO, 2H); 1.25 (m, –**CH$_2$**–, 20H); 0.85 (m, –**CH$_3$**, 3H).

Epoxidation to Epoxy BGx

A 50 mL round-bottom flask with a magnetic stir bar was placed in an oil bath on top of a magnetic stir plate. To the round-bottom flask, 10 g of **BGx** was added, heated to 60 °C, and agitated at 300 rpm. In a 20 mL scintillation vial with a magnetic stir bar placed in an ice bath on top of a magnetic stir plate, glacial acetic acid (7 mL) and >98% sulfuric acid (0.2 mL) were added and agitated at 100 rpm. To the cooled acetic acid, 30 (w/w%) hydrogen peroxide (5 mL) was added dropwise. The solution in the vial was agitated in the ice bath for 20 minutes to allow the formation of the peracetic acid. The peracetic acid solution was then added dropwise (1 drop every 3 seconds) to the brown grease. Upon full addition of the peracetic acid solution, the resulting reaction mixture was allowed to agitate at 300 rpm at 60 °C for 6 hours. Upon completion of the reaction, the reaction was allowed to acclimate to room temperature. To the reaction flask, 50 mL of an aqueous solution of 5 wt% sodium bicarbonate and 5 wt% sodium sulfite was added slowly and agitated. The epoxidized product was then extracted twice with 50 mL of ethyl acetate. The organic phase was then collected, dried under magnesium sulfate for 15 minutes, vacuum-filtered to remove salts, and concentrated under reduced pressure to afford a brown oil that solidified into a wax upon cooling down to room temperature.

Characterization of Epoxy BGc

80% yield. Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 2.34 (m, –CH$_2$–CO, 2H); 1.63 (m, –CH$_2$–CCOO, not determined); 1.25 (m, –CH$_2$–, 21H); 0.85 (m, –CH$_3$, 3H).

Characterization of Epoxy BGb

81% yield. Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 2.33 (m, –CH$_2$–CO, 2H); 1.63 (m, –CH$_2$–CCOO, not determined); 1.25 (m, –CH$_2$–, 21H); 0.85 (m, –CH$_3$, 3H).

Hydroxylation to Hydro BGx

A 100 mL round-bottom flask equipped with a magnetic stir bar was placed in an oil bath on top of a magnetic stir plate. To the flask, 10 g **BGx** was added, heated to 60 °C, and agitated at 300 rpm. In a 20 mL scintillation vial containing a magnetic stir bar, formic acid (0.4 mL) was added and cooled in an ice bath with agitation at 100 rpm. To the cooled formic acid, 30 (w/w%) hydrogen peroxide (10 mL) was added dropwise over the course of 5 minutes. The solution was allowed to stir in the ice bath for an additional 5 minutes before being removed and stirred at room temperature for 15 minutes to allow the *in situ* formation of performic acid. The resulting performic acid solution was then added dropwise (1 drop every 3 seconds) to the pre-heated **BGx** with continuous agitation at 60 °C and 300 rpm. Upon full addition, the reaction mixture was allowed to stir at 60 °C for 16 hours under ambient atmosphere. After completion, the reaction was allowed to cool to room temperature. To the flask, 50 mL of an aqueous solution of 5 wt% sodium bicarbonate and 5 wt% sodium sulfite was added slowly and agitated to neutralize residual acids and quench excess peroxide. The hydroxylated product was extracted twice with 50 mL portions of ethyl acetate. The combined organic extracts were dried over anhydrous magnesium sulfate for 15 minutes, vacuum-filtered to remove residual salts, and concentrated under reduced pressure to yield a viscous brown oil.

Characterization of Hydro BGc

80% yield. Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 2.34 (m, –CH$_2$–CO, 2H); 1.63 (m, –CH$_2$–CCOO, not determined); 1.49 (m, CH–OH, not determined); 1.25 (m, –CH$_2$–, 21H); 0.85 (m, –CH$_3$, 3H).

Characterization of Hydro BGb

80% yield. Proton NMR [300 MHz, CDCl$_3$, δ (ppm)]: 2.33 (m, –CH$_2$–CO, 2H); 1.63 (m, –CH$_2$–CCOO, not determined); 1.25 (m, –CH$_2$–, 22H); 0.85 (m, –CH$_3$, 3H).

Author contributions

Conceptualization, R. C. S.; methodology, R. C. S.; formal analysis, Y. B. K.; investigation, Y. B. K.; resources, R. C. S.; data curation, Y. B. K.; writing—original draft preparation, R. C. S and Y. B. K.; writing—review and editing, all authors; supervision, A. D. S. and R. C. S.; funding acquisition, A. D. S. and R. C. S. All authors have read and agreed to the published version of the manuscript.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: TGA curves, IR, and ^{1}H NMR spectrum provided. See DOI: https://doi.org/10.1039/d5ra07581e.

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