BIOLUBRICANT SYNTHESIS AND CHARACTERIZATION

Umesh Chandra Sharma, PhD Department of Chemical Engineering University Institute of Engineering and Technology C S J M University, Kanpur-208024

Lubrication

Lubrication is the act of applying lubricating substances that are capable of reducing friction between moving mechanical parts



Dr. Umesh Chandra Sharma / UIET, Kanpur

Lubricant

• A lubricant is a substance in the form of a liquid oil / semi-solid grease applied between two metal surfaces in relative mutual motion to prevent their direct contact and thus reduce friction and wear between them.



Functions of a lubricant

- To maintain the moving components of a mechanical device in design condition as long as possible
- Removal of contaminants and wear particles from the metal surfaces
- Dissipation of heat from the mechanical device during operation
- Prevention of rust and corrosion
- To provide a seal at moving contacts to prevent leakage

Global lubricant market

Table 1: Annual global demand for lubricants				
Year	Demand (MMTPA)	Year	Demand (MMTPA)	
2000	36.4	2009	32.2	
2001	35.6	2010	34.5	
2002	35.7	2011	35.1	
2003	35.4	2012	35.0	
2004	36.1	2013	35.3	
2005	36.5	2014	35.4	
2006	36.9	2015	35.6	
2007	36.0	2016	35.7	
2008	36.0	2017	36.1	

Dr. Umesh Chandra Sharma / UIET, Kanpur

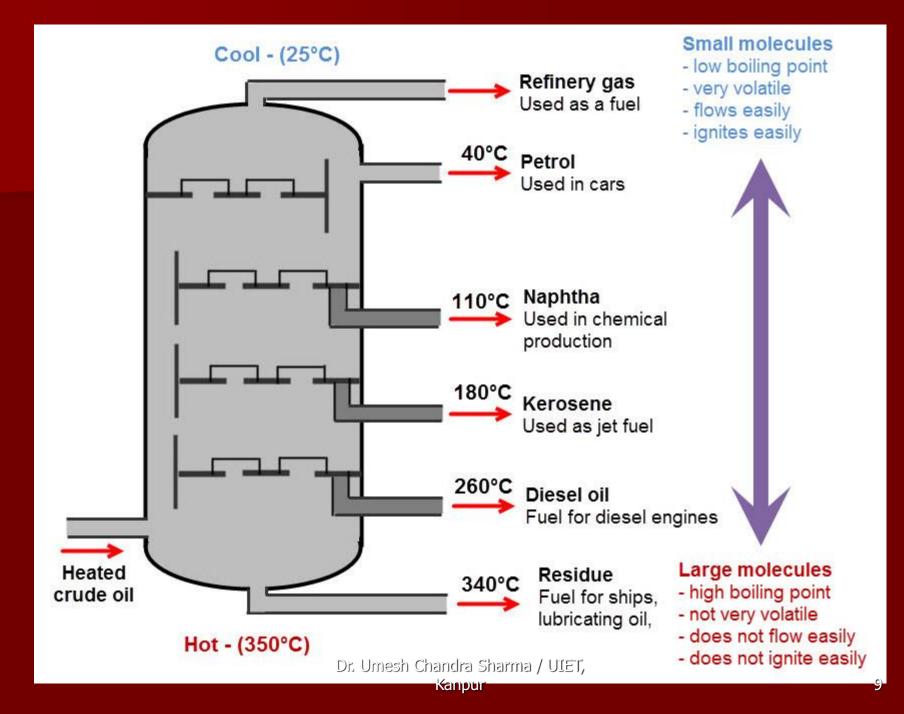
Types of lubricant basestocks

- Basestocks for lubricant formulation
 - Petroleum based mineral oil
 - Vegetable oil or animal fat
 - Synthetic type

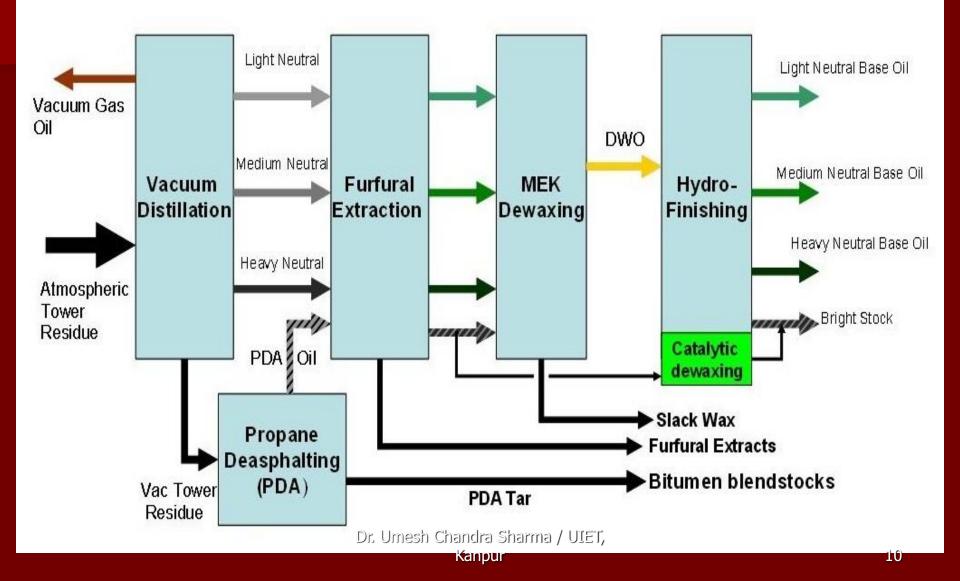
Mineral oil base stock

Mineral oils are highly complex mixtures of C₂₀-C₅₀ hydrocarbons in boiling range of 300-370°C containing a range of linear and branched alkanes (paraffins), cycloalkanes (naphthenes), alkenes (olefins) and aromatics plus a significant amount of non-hydrocarbon sulphur compounds.

- Mineral oil basestocks are derived from crude petroleum in a series of refining operations involving high vacuum distillation, solvent deasphalting, solvent extraction, solvent or catalytic dewaxing and hydrofinishing processes.
- Mineral oils accounted for 95% of global lube oil basestock demand in 2014. Synthetics and biolubes basestocks respectively accounted for 4% and 1% of global basestock demand during this period.



Lube Oil Refinery Process Flow



Vegetable oil or animal fat

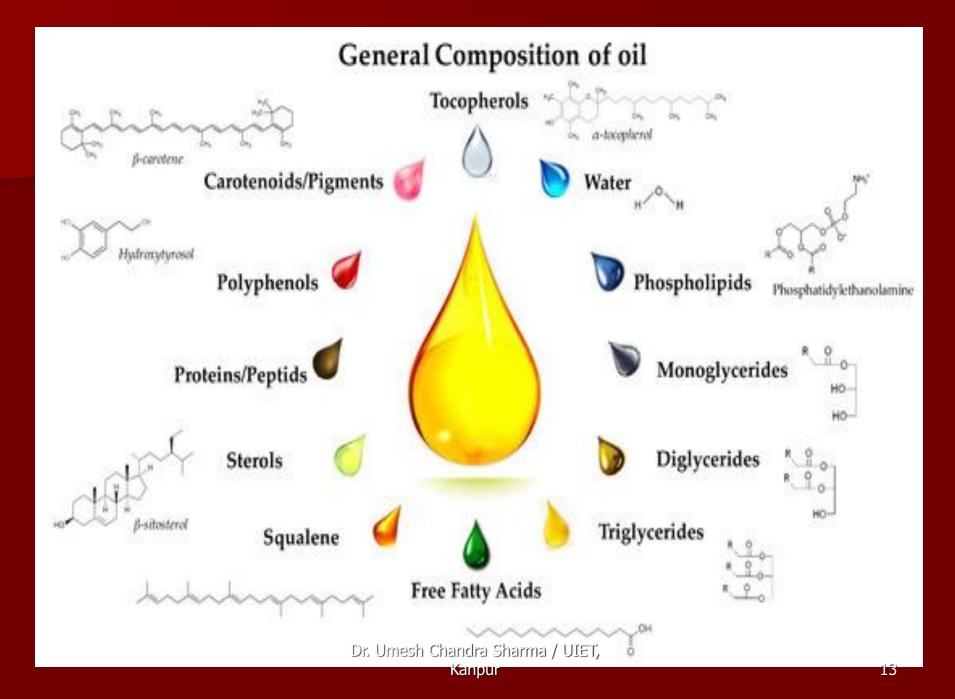
FATS AND OILS



Dr. Umesh Chandra Sharma / UIET,

Kanpur

- Natural oils and fats are amphiphilic compounds of vegetable or animal origin which consist predominantly of
- triglycerides (98%) and
- minor amounts of mono- and di-glycerides (0.5%),
- free fatty acids (0.1%),
- \circ sterols (0.3%) and
- \circ to copherols (0.1%).



- Most fatty acids in natural oils and fats are long chain (C₁₈-C₂₄) linear aliphatic acids (R-COOH).
- The small linear chain fatty acids (C₆) are hydrophilic due to presence of polar water-soluble carboxyl (–COOH) group, whereas long chain fatty acids are increasingly higher hydrophobic (or lipophilic) due to presence of non-polar water-insoluble long hydrocarbon chain.

- The value and application of an oil are determined largely by its fatty acid (FA) composition or the carbon chain distribution.
- The chain length range of C₁₂ (*e.g.* lauric acid) provides important raw materials for detergents and surfactants.
- The chain lengths of C₁₈–C₂₂ (*e.g.* oleic and erucic acid) are used mainly in industrial applications such as lubrication and polymer additives.

Synthetic type basestock

 Synthetic basestocks are chemically engineered by combining low molecularweight compounds in a chemical reaction carried under controlled reaction parameters to produce high molecular-weight compounds of uniform consistency and tailored performance properties. Classification of synthetic basestocks:

- Synthetic Hydrocarbons (*e.g.* polyalphaolefins, alkylated aromatics, polybutenes)
- Synthetic organic esters (*e.g.* aliphatic diesters, polyol esters, phosphate esters)
- Others (*e.g.* polyalkyleneglycols, silicones, *etc.*)

Problems with mineral oil based lubricants

- Depleting crude oil reserves
- Fluctuating oil prices
- Lack of biodegradability and
- Adverse effects on health, safety and environment

- The current supply of crude oil is not going to last indefinitely.
- The researchers may argue over life of existing crude oil resource base; whether it will be for another 100 years or 200 years or so, but there is a general consensus that sources of crude oil will exhaust sooner or later.
- Theory of resource depletion is strengthened by the fact that production rate from the existing fields is declining and new reserves are not being found at the same rate.

- The total quantity of lubricants lost to the environment every year has been estimated at about 50% or approximately 10–15 MMTPA worldwide nearly the same in properties and appearance.
- In addition, significant quantities can enter the biosphere through leakage or failures of seals, pipes or cylinders, quantity remained in filters and containers, incidental or purposive spillage, industrial and municipal discharge, hydraulic and cutting fluids and accidents.

- Mineral oil based lubricants present environmental hazards of the same severity as that displayed by general petroleum.
- These lubricants are poorly biodegradable and toxic and pose serious threats to health, safety and environment at each stage of their production, use and final disposal.
- Possibilities include air pollution, surface and groundwater contamination, soil contamination, and crops and food contamination.

What is a biolubricant ?

- The term biolubricant applies to all lubricants which are both rapidly biodegradable and nontoxic for humans and aquatic environments.
- A biolubricant can be:
 - vegetable oil-based (e.g. soybean oil, rapeseed oil),
 - based on synthetic esters manufactured from modified renewal oils.

Advantages of biolubricants

- Low toxicity
- Lower emissions
- Lower evaporation
- High flash point
- High viscosity index
- High biodegradability
- Better lubricating properties
- Improved user safety
- Improved fuel economy
- Increased equipment life
- Prevention of environmental pollution risk.



Biolubricant feedstock

- Edible vegetable oils (Canola, Coconut, Cottonseed, Groundnut, Palm oil, Rapeseed, Safflower, Sunflower, Soybean, *etc.*)
- Non-edible vegetable oils (Babassu, Castor, *Jatropha*, Jojoba, Karanja, Linseed, Mahua, Neem, Rice bran, Rubber seed, Sesame, Tobacco seed, *etc*.)
- Waste cooking oils
- Animal fats (Chicken fat, Fish oil, Lard, Tallow, *etc.*)

Literature survey

Research group	Base oil	Conclusion
Sivasankaran <i>et al</i> .	Jojoba oil	Good base oil for two-stroke gasoline
(1988)		engine lubricant
Wu et al. (2000)	Rapeseed oil	Epoxidized oil has better friction
		reducing and extreme pressure
		abilities
Fox and Stachowiak	Sunflower	An improvement in the coefficient of
(2003)	oil	friction
Jayadas et al. (2007)	Coconut oil	Good for use as two-stroke engine
		lubricant
Ozioko (2012)	Soybean oil	Good base as a biolubricant
Hamid <i>et al</i> . (2012)	Palm oil	Palm oil based polyol esters as
		potential biolubricant
Dr. Umesh Chandra Sharma / UIET, Kanpur 25		

Research group	Base oil	Conclusion		
Resul et al. (2012)	Jatropha curcas oil	Properties comparable to Palm		
		oil and Soybean based		
		biolubricants		
Madankar et al. (2013)	Canola oil	Epoxy canola is a good		
		starting material for lubricant		
		formulation		
Owolabi <i>et al</i> . (2013)	Water-melon, milk	Promising candidate, but		
	bush and pawpaw	future work is required		
	seed oils			
Sharma <i>et al</i> . (2018)	Karanja oil	Good viscosity-temperature		
		properties		
Borugadda and Goud	Waste cooking oil	Improved physicochemical,		
(2014, 2016)		thermal and rheological		
		properties		
Dr. Umesh Chandra Sharma / UIET,				

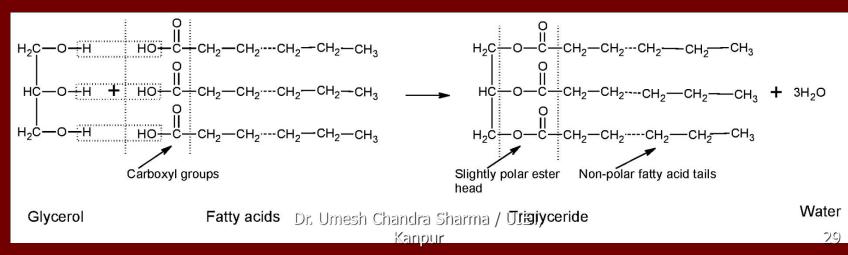
Research group	Base oil	Conclusion
Rani <i>et al.</i> (2015)	Rice bran oil	Good frictional
		properties, but WSD is
		more
Dormo <i>et al.</i> (2004)	Fusel oil plus oleic acid	An environment friendly
		product
Salih <i>et al</i> . (2011)	Oleic acid based esters	Resulting ester can be
		used as biolubricant,
		surfactant, or fuel
Salimon <i>et al</i> . (2012)	Ricinoleic acid	Resulting ester can be
		used as biolubricant,
		surfactant, or fuel
Padmaja <i>et al</i> . (2012)	10-undecenoic acid	Good thermal stability
	based polyol esters	above 260°C
	Dr. Umesh Chandra Sharma / UIET, Kanpur	temperature

SYNTHESIS OF BIOLUBRICANT FROM KARANJA OIL AND BASIC PHYSICOCHEMICAL PROPERTIES EVALUATION

> Dr. Umesh Chandra Sharma / UIET, Kanpur

Structure of vegetable oils

- The greatest part of vegetable oils are triglycerides (98%).
- A triglyceride is the reaction product of one molecule of glycerol and three molecules of fatty acids to produce three molecules of water with one molecule of a triglyceride.



Amphiphilic nature of vegetable oils

- The oils and fats are amphiphilic in nature because they contain both slightly polar hydrophilic ester group and non-polar lipophilic fatty acid chains.
- The polar ester heads of triglyceride molecules attach to the metal surfaces to be lubricated forming a monolayer film.
- The non-polar fatty acid chains stick away from the metal surfaces offering a sliding surface to prevent the metal surfaces to come in direct contact.
- The vegetable oils, therefore, can be used as both boundary and hydrodynamic lubricants.

Vegetable oils advantages

- Biodegradable and non-toxic
- Good boundary lubrication properties
- Excellent thin film strength due to adherence to the surface of metals
- Low volatility
- Higher flash and fire points
- Naturally high viscosity index



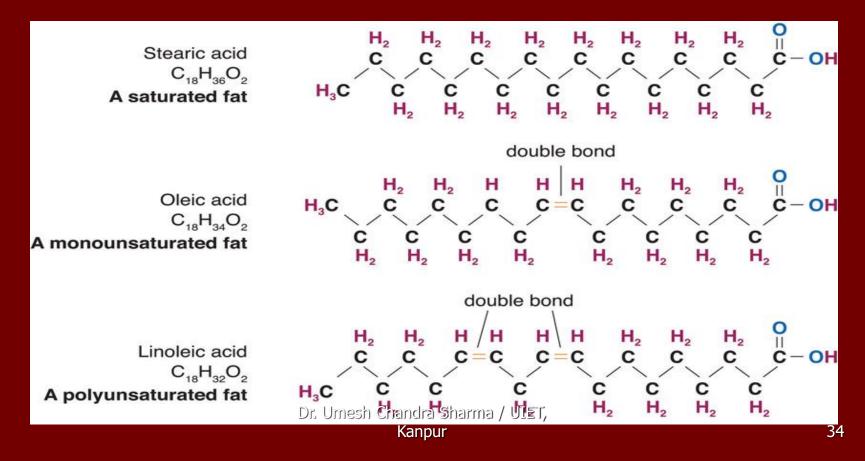
Vegetable oils disadvantages

- Restricted industrial use due to poor thermal and oxidation stability
- Low cold flow behaviour
- Higher prices
- Shorter life and storage time
- Poor seal compatibility (need modifications)

Chemical modification methods

- Most vegetable oils do not contain hydroxyl groups in their structure (castor and lesquerella oils are only known exceptions).
- Biolubricant synthesis from vegetable oils requires the introduction of hydroxyl groups in oil structures by a suitable chemical modification method.
- Various methods are Transesterification, selective hydrogenation at unsaturated sites, structural modification by *in-situ* epoxidation, catalytic cracking, catalytic esterification, emulsification, additive treatment and genetic modification.

- The unsaturated C=C double bonds and ester linkages are two main moieties in vegetable oil structures.
- All chemical modification methods for biolubricant production start from one of these two moieties.



- Transesterification is the most adopted chemical modification route and Trimethylolpropane (TMP) is the generally used polyhydric alcohol.
- Transesterification reduces the β-hydrogen from the triglyceride molecule and thus improves the thermo-oxidative stability of resulting triester, which is an important property for any lubricant.
- TMP is cheaper than other polyhydric alcohols and reacts at low temperature due to its low melting point, hence TMP is more economical than other polyhydric alcohols.

Chemical structure of biodiesel

Biodiesel is a mixture of mono alkyl (methyl/ethyl) esters of long chain fatty acids between 16 and 22 carbon atoms like lauric, palmitic, stearic, oleic, *etc.* derived from vegetable oils or animal fats.

 Biodiesel is conventionally obtained in a chemical process by transesterification of vegetable oils or animal fats with short and linear carbon chain alcohols such as methanol or ethanol under catalytic effect of sodium/potassium hydroxide. ■ Karanja oil + Methanol + catalyst → Karanja oil methyl ester (KOME) + Glycerin

Biodiesel thus produced is a precursor for synthesis of polyol ester biolubricants.

Around 1 kg of glycerol is produced as a byproduct for every 10 kg of biodiesel produced by transesterification process.

Chemical structure of biolubricant

The prepared biodiesel (fatty acid mono alkyl esters) are transesterified with branched polyhydric alcohols such as neopentyl glycol (NPG), trimethylolpropane (TMP) or pentaerythritol (PE) using sulphuric acid as catalyst to form polyol ester biolubricants.

• KOME + Trimethylol propane (TMP) + catalyst \rightarrow Biolube

Polyol esters contain at least one quaternary carbon atom that imparts higher chemical stability to the molecules as compared to glyceryl esters.

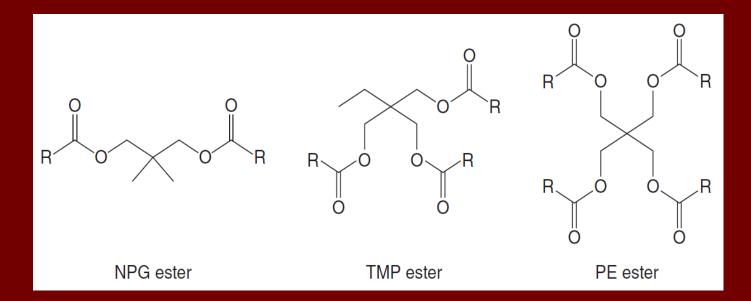


Figure: Polyol ester biolubricants prepared using different polyols Dr. Umesh Chandra Sharma / UIET, Kanpur

Karanja oil as feedstock

- Scientific name Millettia pinnata (earlier known as Pongamia pinnata)
- Found in parts of Indian subcontinent, China, Japan, Malaysia, Australia, Pacific islands, Indonesia, Philippines, United States, Fiji, New Zealand and Seychelles.
- In India the Karanja seeds are found in A.P., Bihar, Chhatisgarh, Jharkhand, M.P., Kerala, Karnataka, Maharashtra, Orissa and W. Bengal.

- Karanja is a fast growing medium sized leguminous tree of about 18 m height with a trunk diameter over 50 cm.
- It is an evergreen nitrogen-fixing tree that is highly tolerant to harsh climatic conditions like drought, heat, frost, salinity, water logging, *etc.* and hence can be easily grown on most soil types ranging from clay to sandy to stony without much care.



■ The tree starts bearing fruits at the age of 6-7 years. The pods are elliptical, 2-3 cm wide, 3-7.5 cm long, 0.1-1.5 cm thick and contain single light brown seed of 10-28 mm length. The seeds contain 27-39% by weight of oil that is yellowish orange to brown when freshly extracted and rapidly darkens on storage. The oil mostly contains monounsaturated fatty acids (66.6%) with rest as saturated (15.8%)and polyunsaturated (16.7%) fatty acids.

Dr. Umesh Chandra Sharma / UIET, Kanpur

5

Dr. Umesh Chandra Sharma / UIET, Kanpur





- Several toxic flavonoids including karanjin (1.25%), kanjone, pongamol (0.85%), pongapin, pinnatin, pongagalabrone, pongaglabrin, glabrachalcone, isopongachromene, *etc.* are responsible for disagreeable taste and odour of KO, which makes the oil non-edible.
- National Oilseeds and Vegetable Oil
 Development (NOVOD) Board has estimated the potential of oil in India at about 55,000 tonnes/year; of this only 6% is currently used.

 Karanja oil has been traditionally used for soap making, leather tanning, illumination and medicinal purposes.

Its use as a raw material for biodiesel and biolubricant production is on rise since past few decades.

Properties of Karanja oil

- Karanja oil was first analysed for its physicochemical properties prior to synthesis of KOME biodiesel.
- Indian Standard Methods of Sampling and Test for Oils and Fats Part I Sampling, Physical and Chemical Tests (Revised) IS: 548 (Part I) – 1964 was followed for all tests.

- Acid value and saponification value of base oil are specifically important for the purpose of chemical modification.
- The acid value of source oil should be under 2 mg KOH/g triglyceride (equivalent to 1% free fatty acid) for direct base-catalysed transesterification.
- If not, excess acid value is reduced by a pretreatment process mostly acid-catalysed esterification of high free fatty acid containing vegetable oil.

Similarly, the saponification value provides the estimate of molar mass of test compound molecule.

The molar mass of triglyceride or methyl ester molecule is required to determine the stoichiometric quantities of other reactants participating in esterification and transesterification reactions.

Table 2: Analysis of Karanja oil used in study						
S. No.	Parameter	Value				
1.	Density (gm/ml)	0.9088 (27°C)				
2.	Acid value (mg KOH/gm)	8.54				
3.	Free fatty acid (FFA)	4.27% by weight				
4.	Colour (1/4 inch)	20 Y + 3.1 R [Y = 1, R = 5]				
5.	Moisture index (%w/w)	0.17				
6.	Refractive index	1.4770 (40°C)				
7.	Saponification value (mg KOH/gm)	189.8				
8.	Iodine value (gm $I_2/100$ gm)	87.2				
9.	Unsaponifiable matter (%w/w)	2.1				
Dr. Umesh Chandra Sharma / UIET, Kanpur 52						

Table 3: Fatty acid composition of Karanja oil							
Fatty acid	Chemical name	Formula	Structure	Weight%			
Palmitic	Hexadecanoic	$C_{16}H_{32}O_2$	16:0	10.6			
Stearic	Octadecanoic	$C_{18}H_{36}O_2$	18:0	6.8			
Oleic	Cis-9-octadecenoic	$C_{18}H_{34}O_2$	18:1	49.4			
Linoleic	Cis-9, cis-12- octadecenoic	C ₁₈ H ₃₂ O ₂	18:2	19.0			
Arachidic	Eicosanoic	$C_{20}H_{40}O_2$	20:0	4.1			
Behenic	Docosanoic	$C_{22}H_{44}O_2$	22:0	5.3			
Lignoceric	Tetracosanoic	$C_{24}H_{48}O_2$	24:0	2.4			

Table 4: Properties of reactants							
Material	M.W.	Density	M.P./B.P.	Flash point			
	(g/mol)	(g/ml)	(°C)	(°C)			
Karanja oil	886.71	0.9088					
Methanol	32.04	0.792	/64.7				
Sulphuric acid	98.08	1.84					
Potassium	56.11	2.12					
hydroxide							
TMP ($C_6 H_{14} O_3$)	134.17	1.084	58 / 289	172			

Synthesis of KOME biodiesel

Pretreatment of Karanja oil

Oil was first filtered to remove any solid material; then it was preheated at 110°C for 30 min to remove moisture, as the moisture is responsible for saponification in the reaction.

Acid/Base-catalyzed Transesterification Reaction



Acid-catalyzed Esterification Reaction

- The high FFA content (≥ 1%) in oil is not suitable for base-catalyzed transesterification as it results in excessive soap formation via saponification reaction.
- To reduce the FFA of oil, acid-catalyzed reaction was carried out prior to base-catalyzed reaction.

Reaction:

Karanja oil + Methanol (1: 6 M ratio) + H_2SO_4 catalyst (1.0 wt% of oil) + 60°C temperature + stirring speed 600 rpm + 5 hour time

- The product obtained was allowed to settle overnight in a separating funnel. Two layers were formed: KOME rich bottom layer and methanol-water top layer due to differences in their densities and their polarities.
- KOME rich layer was separated and washed with warm water until the discarded wash water reached a pH level of 6-7.
- Moisture was completely removed from the KOME before the base-catalyzed reaction.

Analysis of oil after Acid-catalyzed Reaction

Acid value
FFA
Saponification Value
Molecular weight

2.16 mg KOH/gm1.08%161.271043.61 g/mol

Base-catalyzed Transesterification Reaction

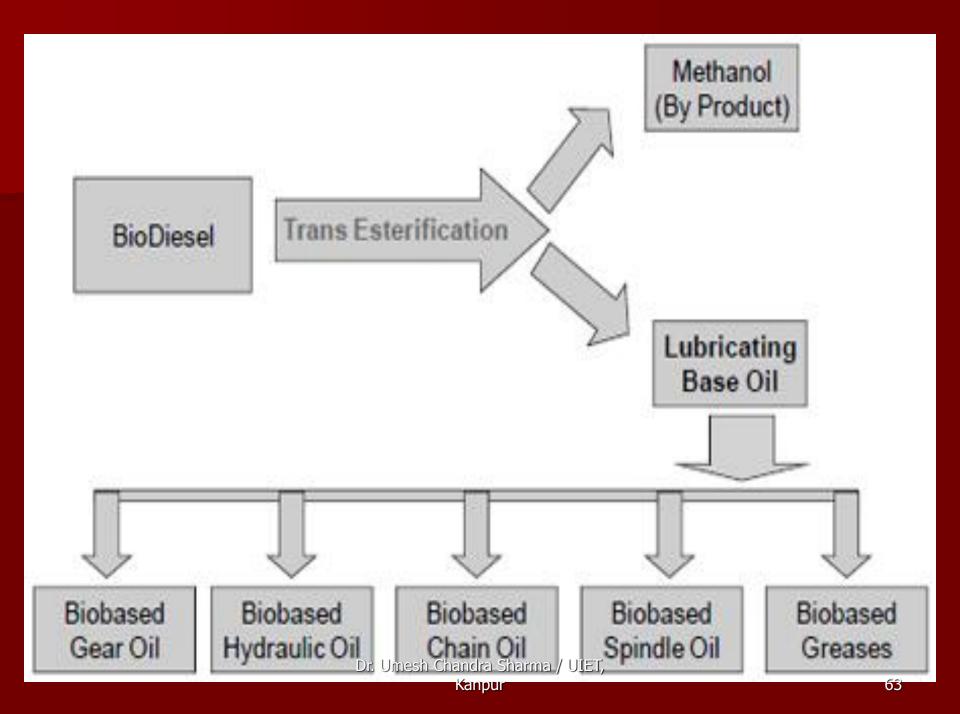
- Acid-catalyzed oil + Methanol (1: 6 M ratio) + KOH catalyst (1.25 wt% of oil) + 65°C temperature + stirring speed 600 rpm + 5 hour time → KOME + glycerin
- Immediately after the reaction, oxalic acid was added to neutralize the remaining catalyst.
- The product obtained was allowed to settle for 24 hours into two phases in a separating funnel.

 Two layers were formed: KOME rich top layer and glycerol rich bottom layer due to differences in their densities and their polarities.

KOME layer was washed with equal volume of warm water to remove any remaining soaps, salts or FFAs and then dried over anhydrous sodium sulfate.

The washing was repeated until the discarded wash water reached a pH level of 6-7 and no soap bubbles appeared in it.

Analysis of KOME (Biodiesel) Acid value 1.64 mg KOH/gm 0.82% **FFA** Saponification Value 149.30 1127.22 Molecular weight ■ Iodine value 190 0.8888 g/ml Density



Biolube Synthesis Reaction



Synthesis of KOTMPE biolubricant Second Stage Transesterification Reaction KOME + TMP (4:1 M ratio) + H_2SO_4 catalyst $(2\% \text{ w/w of methyl ester}) + 3 \text{ hours} + 150^{\circ}\text{C} +$ continuous stirring under 10 mbar vacuum \rightarrow Biolube (Karanja oil based polyol esters) The product obtained was allowed to cool overnight in a separating funnel and then washed with warm water to remove any remaining catalyst.

- The biolube was then dried over anhydrous sodium sulfate to remove any remaining moisture.
- The product obtained was of dark colour due to reaction in strong acidic medium.
- The biolube was bleached with mixture of 9% w/w Fuller's earth and 1% w/w activated carbon at 100°C for 30 min with stirring.

Analysis of Synthesized Biolubricant

- Acid value
- FFA
- Iodine value
- Density
- Flash Point
- Fire Point
- Viscosity at 40°C
- Viscosity at 100°C
- Viscosity index

1.08 mg KOH/gm 0.54%203 0.9208 g/ml 174°C 216°C 58.6 sec (15.2356 cSt) 35.09 sec (9.1228 cSt) 206.718

Table 5: Comparison of properties of KO, KOME andKOTMPE

Property	KO	KOME	КОТМРЕ
Acid value, mg KOH/gm	8.54	1.64	1.08
FFA, %	4.27	0.82	0.54
Saponification value, mg	189.8	149.30	
KOH/gm			
Molecular weight, gm/mol	886.71	1127.22	
Iodine value, gms $I_2/100$ gm oil or	87.2	190	203
fat			
Density, gm/ml@27°C	0.9088	0.8888	0.9208
Viscosity@40°C, cSt Dr. Umesh Char	idra Shanmay VIET acourt	22.83	15.23
		12.00	0.10

KO (1), KOME (2) and KOPTE (3)



Kanpur

VISCOUS FLOW BEHAVIOUR OF SYNTHESIZED BIOLUBRICANT

Dr. Umesh Chandra Sharma / UIET, Kanpur

Need for rheological studies

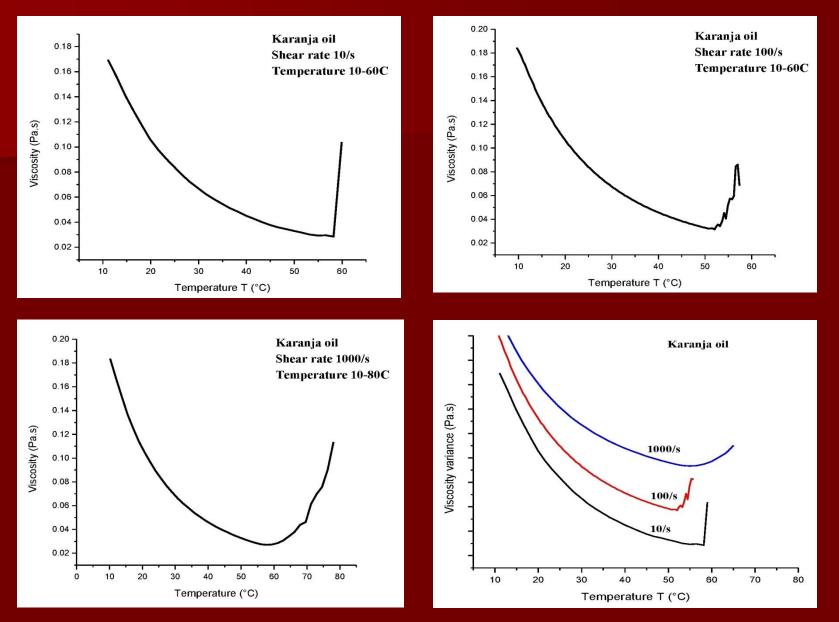
- Rheology is the study of flow and deformation of material under application of stress.
- Knowledge of rheological properties of KO, KOME and KOTMPE is essential in movement of bulk quantities of these oils to have better control over oil velocity, pump pressure, and energy requirement in synthesis process.

- Viscosities of all samples were measured at shear rates 10, 100 and 1000 s⁻¹ in a temperature range of 0°C to 78°C using Discovery Rheometer HR-3 model manufactured by TA instruments.
- System geometry applied for analysis of samples was Cone and plate type with 60 mm dia and ½ degree angle.



Variation of Viscosity with Temperature at Shear Rates 10, 100 and 1000

Table 6: Viscosity variation for Karanja oil with temperature						
Temperature (°C)	Viscosity (Pa·s) at	Viscosity (Pa·s) at	Viscosity (Pa·s) at			
	shear rate 10	shear rate 100	shear rate 1000			
10.000	0.176996	0.181928	0.185292			
15.000	0.138772	0.138243	0.140890			
20.000	0.105482	0.106700	0.108383			
25.000	0.0836515	0.0840197	0.0854306			
30.000	0.0668151	0.0674680	0.0683829			
35.000	0.0544948	0.0552262	0.0557846			
40.000	0.0450114	0.0457363	0.0460993			
45.000	0.0376830	0.0384616	0.0387085			
50.000	0.0329716	0.0329437	0.0327670			
55.000	0.0293690	0.0529011	0.0282555			
60.000	0.1097801		0.0277548			



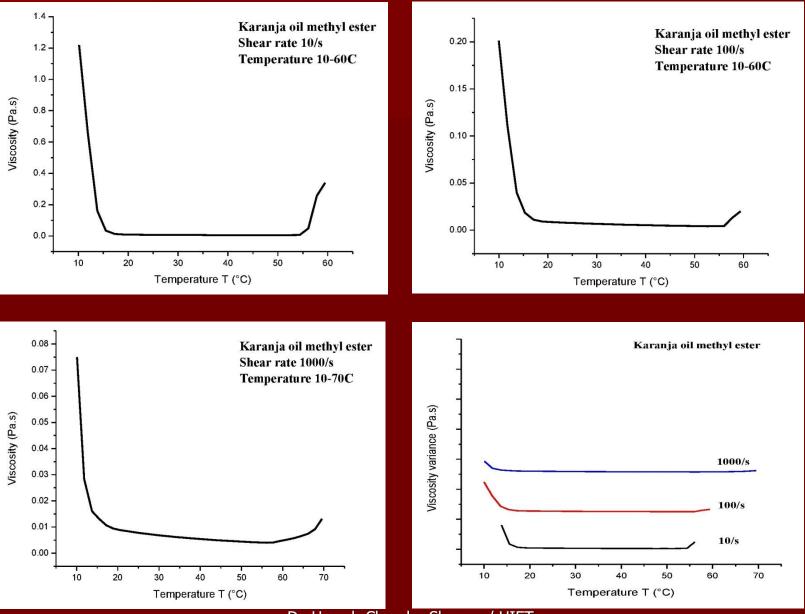
- The viscosity changes for KO at shear rates 10, 100 and 1000 s⁻¹ in temperature range of 10-60/80°C were quite identical with some variation beyond 50°C due to changes in applied shear rates.
- Viscosity decreased exponentially with increase in temperature up to 58°C at shear rate 10 s⁻¹, up to about 51°C at shear rate 100 s⁻¹ and up to about 58°C at shear rate 1000 s⁻¹.
- The viscosity of the oil was found to increase at around 60°C.
- The gain in viscosity was rapid at shear rate 10 s⁻¹ and gradual at shear rates 100 and 1000 s⁻¹.

- The effect of high shear rates on viscosity of oil sample can be easily seen here.
- KO taken for current study contained 4.27% by weight of free fatty acids.
- It is likely that some of low molecular weight free fatty acids started decomposing around 60°C causing an increase in viscosity of oil.
- This increase in viscosity was rapid at low shear rate, but was slow at high shear rates of 100 and 1000 s⁻¹.
- It was possibly due to easy homogenization of oil at high shear rates.

Variation of Viscosity with Temperature at Shear Rates 10, 100 and 1000

Table 7: Viscosity variation for Karanja oil methyl ester with								
	temperature							
Temperature (°C)	emperature (°C) Viscosity (Pa·s) at Viscosity (Pa·s) at Viscosity (P							
	shear rate 10	shear rate 100	shear rate 1000					
10.000	1.27764	0.200564	0.0775210					
15.000	0.073993	0.0225768	0.0138854					
20.000	9.10622e-3	8.79688e-3	9.04068e-3					
25.000	7.59601e-3	7.58696e-3	7.79821e-3					
30.000	6.72088e-3	6.64710e-3	6.84053e-3					
35.000	6.09534e-3	5.88185e-3	6.05473e-3					
40.000	5.59492e-3	5.25794e-3	5.41423e-3					
45.000	5.51082e-3	4.73686e-3	4.87535e-3					
50.000	5.64289e-3	4.30834e-3	4.41866e-3					
55.000	0.0216695	4.22830e-3	4.05195e-3					
60.000	0.367838 Chandre	0.0226480	4.84963e-3					
	Kanr							

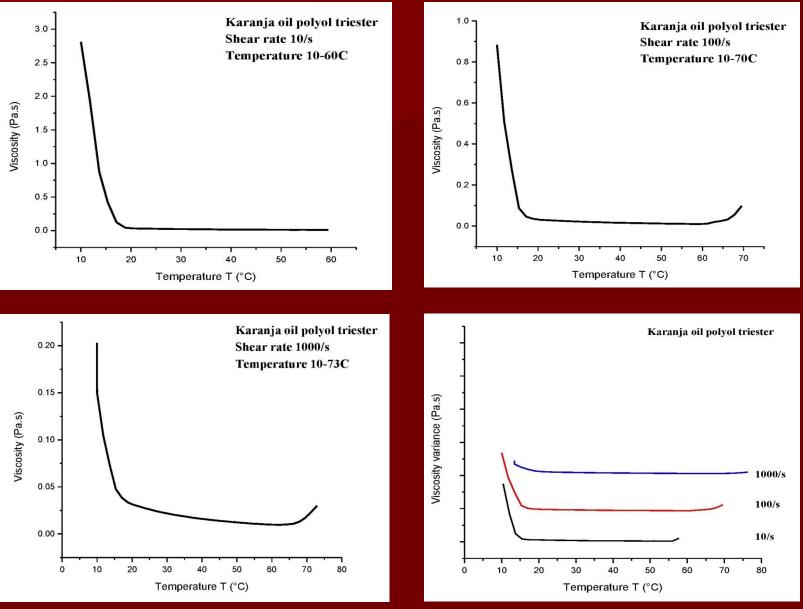
• 4 1



- The viscosity changes for KOME at shear rates 10, 100 and 1000 s⁻¹ in temperature range of 10-60/70°C were almost identical irrespective of applied shear rates.
- First, there was a steep decline in viscosity between 10 and 15°C. Thereafter, viscosity decreased with increase in temperature at a very small rate from 20 to 55°C and finally it started increasing from 55°C.
- The rise in viscosity after 55°C was more at low shear rate and diminished with increasing shear rates due to easy homogenization of oil at high shear rates.

Variation of Viscosity with Temperature at Shear Rates 10, 100 and 1000

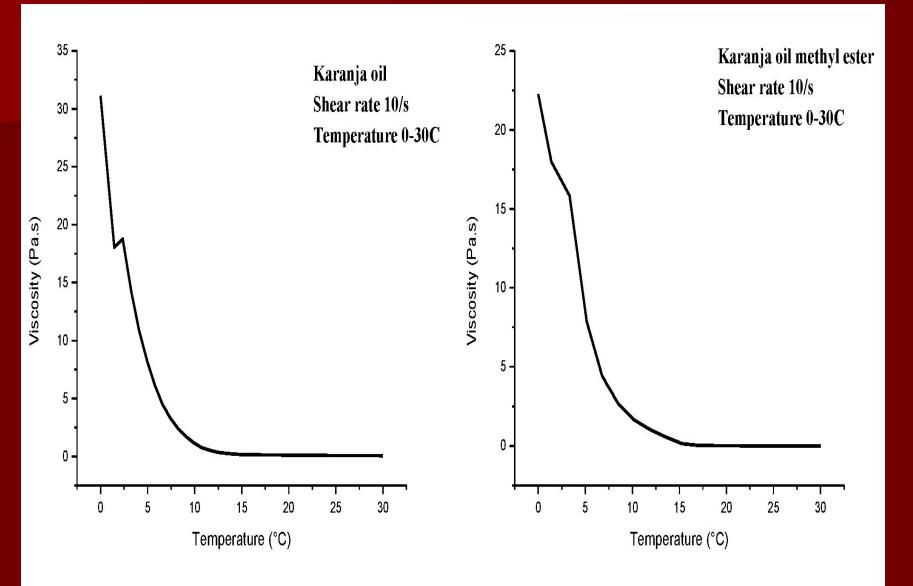
Table 8: Viscosity variation for Karanja oil trimethylolpropane ester with							
	temperature						
Temperature (°C)	Viscosity (Pa·s) at	Viscosity (Pa·s) at					
	shear rate 10	shear rate 100	shear rate 1000				
10.000	2.82079	0.884947	0.149381				
15.000	0.524164	0.123720	0.0529538				
20.000	0.0354434	0.0321982	0.0316178				
25.000	0.0268172	0.0260752	0.0260813				
30.000	0.0223047	0.0218621	0.0218394				
35.000	0.0188464	0.0185797	0.0186024				
40.000	0.0162112	0.0159482	0.0160286				
45.000	0.0140853	0.0138869	0.0139375				
50.000	0.0123833	0.0122207	0.0122479				
55.000	0.0115125	0.0108380	0.0108568				
60.000	0.0120352	0.0105045	9.89712e-3				
65.000		a Sharm a) (1972136	0.0103467				
70.000	Kam; 	0.1095491	0.0187686				



- The viscosity changes for KOTMPE at shear rates 10, 100 and 1000 s⁻¹ in temperature ranges of 10-60/70/73°C were very much identical and followed pattern similar to KOME.
- First, there was a sharp decline in viscosity between 10 and 20°C followed by a slower decrease from 20 to 60/65°C. Then viscosity started increasing from 60/65°C.
- This rise in viscosity was more pronounced at low shear rates due to lesser torque applied on triester sample compared to sample under high shear rates.

Low temperature viscosity behaviour of Karanja oil and Karanja oil methyl ester at Shear rate 10 $\rm s^{-1}$

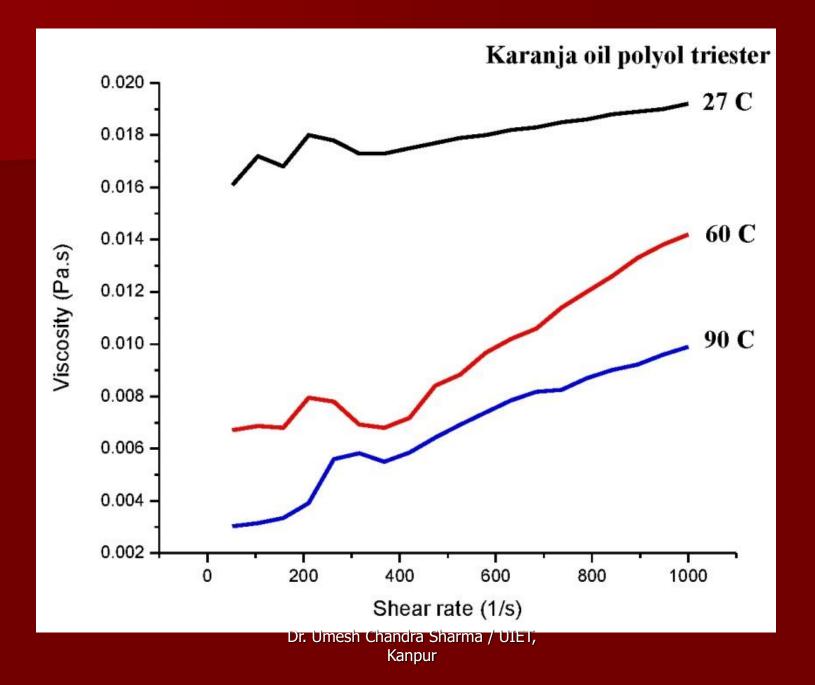
Table 9: Viscosity variation at shear rate 10 s ⁻¹					
Temperature (°C)	Karanja oil	Karanja oil methyl ester			
0.000	31.0898	22.2439			
5.000	8.10398	8.52691			
10.000	1.12067	1.75294			
15.000	0.168213	0.202099			
20.000	0.106458	0.0114692			
25.000	0.0831205	7.75282e-3			
30.000	0.0666476	7.06664e-3			



- Low temperature flow behaviors of KO and KOME were analyzed at 0-30°C.
- The synthesized biolubricant KOTMPE was not analyzed below 10°C as the pour point of the sample was 6°C and the triester sample got solidified at low temperature.
- The rheological behaviors of KO and KOME in low temperature range of 0-30°C were nearly identical.
- The viscosity decreased sharply from 0 to 10°C in KO and from 0 to 15°C in KOME.
- Thereafter, it became constant for remaining temperature range.

Variation of Viscosity with Shear Rate for KOTMPE

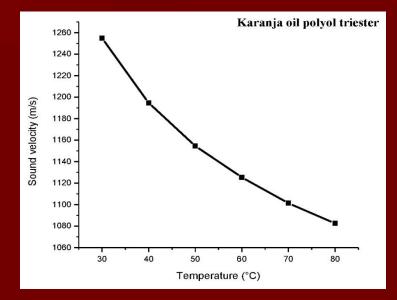
Table 10: Variation of Viscosity with Shear Rate for KOTMPE					
Shear Rate (1/s)	Viscosity (Pa·s)				
	at 27°C	at 60°C	at 90°C		
100	0.0170	0.00685	0.00314		
150	0.0168	0.00681	0.00332		
200	0.0177	0.00771	0.00380		
250	0.0178	0.00783	0.00518		
300	0.0174	0.00719	0.00576		
350	0.0174	0.00684	0.00561		
400	0.0174	0.00703	0.00571		
450	0.0176	0.00785	0.00617		
500	0.0178	0.00862	0.00668		
550	0.0179	0.00921	0.00713		
600	0.0180	0.00988	0.00757		
650	0.0182	0.0103	0.00796		
700	0.0183	0.0108	0.00820		
750	0.0185	0.0116	0.00837		
800	0.0186	0.0121	0.00876		
850	0.0188	0.0127	0.00904		
900	0.0189	0.0133	0.00925		
950	0.0190	0.0138	0.00961		
1000	DP.0793		0.0099		



- The viscosity variations for synthesized biolubricant KOTMPE with shear rates at 27, 60 and 90°C were analyzed.
- Shear rates were varied from 100 to 1000 s⁻¹.
- It was found that viscosities of biolubricant sample increase with shear rates at a given temperature.
- The results expressed a shear rate thickening behaviour for synthesized biolubricant, which is a desirable property for high temperature and high shear application lubricants as the lubricant will not thin out between moving engine parts at high temperature and high shear rate.

Variation of ultrasonic velocity with temperature

Table 11: Sound velocity variation				
Temperature (°C)	Sound Velocity (m/s)			
30	1.25482×10^{3}			
40	1.19468×10^{3}			
50	1.15459×10^{3}			
60	1.12542×10^{3}			
70	1.10126×10^{3}			
80	1.08261×10^{3}			



Sound velocity was higher at low temperatures. As the temperature increased, the ultrasonic velocity decreased, which demonstrated the weakening of intermolecular forces due to thermal agitation.

Application of Arrhenius Equation and Power Law Model For Flow Characterization of Synthesized Biolubricant

The synthesized biolubricant was analyzed for its viscosity in temperature range of 10–60°C at shear rates of 10, 100, and 1000 s⁻¹.

These data were then fitted in Arrhenius equation and Ostwald-deWaele power law model for flow characterization.

Viscosity variation for synthesized biolubricant with temperature at different shear rates

Table 12: Viscosity variation for Karanja oil trimethylolpropane

ester						
Temperature (°C)	Viscosity (Pa·s) at	Viscosity (Pa·s) at	Viscosity (Pa·s) at			
	shear rate 10	shear rate 100	shear rate 1000			
10.000	2.82079	0.884947	0.149381			
15.000	0.524164	0.123720	0.0529538			
20.000	0.0354434	0.0321982	0.0316178			
25.000	0.0268172	0.0260752	0.0260813			
30.000	0.0223047	0.0218621	0.0218394			
35.000	0.0188464	0.0185797	0.0186024			
40.000	0.0162112	0.0159482	0.0160286			
45.000	0.0140853	0.0138869	0.0139375			
50.000	0.0123833	0.0122207	0.0122479			
55.000	0.0115125	0.0108380	0.0108568			
60.000	Dr. Umesh Chandra 0.0120352 Kanr	a Sharma / ULET, Jur 0.0105045	0.0098971			

The effect of temperature on apparent viscosity of the biolubricant was articulated by the following Arrhenius type equation:

$$\mu = A \exp(E_a/RT) \qquad \dots (1)$$

where A = frequency factor (Pa.s) $E_a = activation energy (J mol^{-1})$ R = gas constant (8.314 J mol^{-1} K^{-1}) and T = absolute temperature (K).

- A plot of ln (*apparent viscosity*) versus 1/(*absolute temperature*) was constructed for test sample at each shear rate.
- As the cold flow behavior of biolubricant sample was quite different from that at higher temperatures, separate plots were drawn for temperature ranges 10–20°C and 20–60°C.
- A best fit trend line was drawn by connecting the actual data points. Then the slope, intercept and regression coefficient were calculated with this line.

- Activation energy *E_a* was calculated by multiplying the slope of the straight line to the gas constant *R*, and *A* was an exponential of the intercept.
- The activation energy represents a sort of energy barrier that should be overpowered for an elementary flow process to proceed.
- The biolubricant viscosities were predicted using the Arrhenius equation at 10, 20, 30, 40, 50, and 60°C temperature and shear rate variation of 10, 100, and 1000 per second.

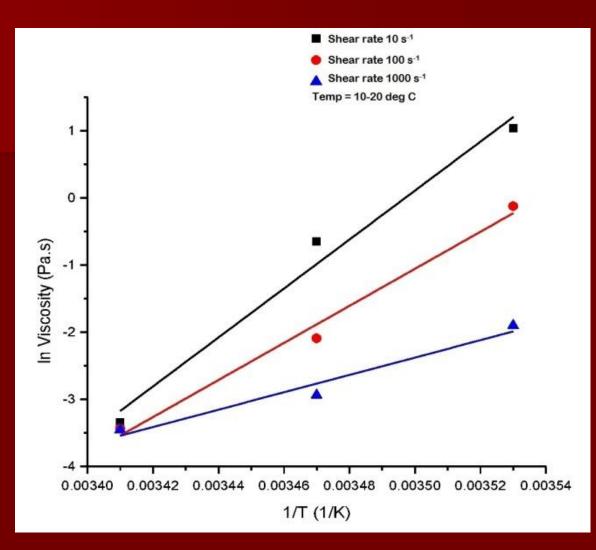


Figure: Arrhenius plot for apparent viscosity of biolubricant oil in temperature range of 10-20°C

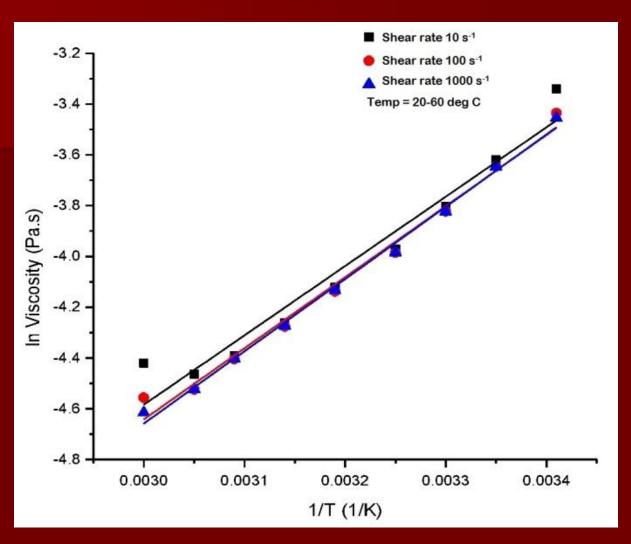


Figure: Arrhenius plot for apparent viscosity of biolubricant oil in temperature range of 20-60°C

The Arrhenius equation parameters obtained from two plots are given below in Table 13.

Tab	Table 13: Arrhenius parameters for synthesized biolubricant oil						
γ (1/s)	$\mathbf{T} = 10 \mathbf{-20^{\circ}C}$			$\mathbf{T} = 20 \mathbf{-60^{\circ}C}$			
	A (Pa.s)	E _a (J/mol)	R ²	A (Pa.s)	E _a (J/mol)	R ²	
10/s	4.05 × 10 ⁻⁵⁶	303,242.06	0.9651	2.82 × 10 ⁻⁶	22,706.42	0.9453	
100/s	3.71 × 10 ⁻⁴³	229,577.95	0.9768	2.17 × 10 ⁻⁶	23,274.68	0.9857	
1000/s	1.99 × 10 ⁻²¹	107,581.77	0.9276	1.86 × 10 ⁻⁶	23,653.21	0.9949	

The predicted viscosity values agreed well with the measured viscosity values as evident from the given Table 14.

Table 14: Me	Table 14: Measured and predicted (in parenthesis) viscosity data in Pa.s					
	at differ	ent shear rates				
Temp (°C)	Shear rate 10/s	Shear rate 100/s	Shear rate 1000/s			
10.00	2.821 (3.804)	0.885 (0.881)	0.149 (0.143)			
20.00	0.035 (0.047)	0.032 (0.032)	0.032 (0.030)			
20.00	0.035 (0.032)	0.032 (0.031)	0.032 (0.031)			
30.00	0.022 (0.023)	0.022 (0.022)	0.022 (0.022)			
40.00	0.016 (0.017)	0.016 (0.017)	0.016 (0.016)			
50.00	0.012 (0.013)	0.012 (0.013)	0.012 (0.012)			
60.00	0.012 (0.010)	0.011 (0.010)	0.010 (0.010)			

- The high magnitude of R² value indicated that changes in apparent viscosity of synthesized biolubricant with temperature could be modelled suitably using the Arrhenius equation.
- The study confirmed that the viscosity of the synthesized biolubricant was largely influenced by temperature; however, this effect was more pronounced at low temperature and was moderate at high temperatures.

Shear stress calculation at varying shear rates

The shear stress values corresponding to a given shear rate were obtained by multiplying the viscosity with shear rate at 27°C, 60°C, and 90°C temperatures following relation:

 $\tau = \mu \gamma$ (2) Where $\tau =$ shear stress (Pa) $\mu =$ dynamic viscosity (Pa.s) $\gamma =$ shear rate (s⁻¹)

Table 15	Table 15: Viscosity and shear stress versus shear rate measurements at different					
			temperatures			
Shear rate		Viscosity (Pa·s))	S	Shear stress (Pa	a)
(1/s)	27°C	60°C	90°C	27°C	60°C	90°C
100	0.0244	0.01050	0.00314	2.44	1.05	0.31
150	0.0242	0.01046	0.00332	3.63	1.57	0.50
200	0.0251	0.01136	0.00380	5.02	2.27	0.76
250	0.0252	0.01148	0.00518	6.30	2.87	1.30
300	0.0248	0.01084	0.00576	7.44	3.25	1.73
350	0.0248	0.01049	0.00561	8.68	3.67	1.96
400	0.0248	0.01068	0.00571	9.92	4.27	2.28
450	0.0250	0.01150	0.00617	11.25	5.18	2.78
500	0.0252	0.01227	0.00668	12.60	6.14	3.34
550	0.0253	0.01286	0.00713	13.92	7.07	3.92
600	0.0254	0.01353	0.00757	15.24	8.12	4.54
650	0.0256	0.01395	0.00796	16.64	9.07	5.17
700	0.0257	0.01445	0.00820	17.99	10.12	5.74
750	0.0259	0.01525	0.00837	19.43	11.44	6.28
800	0.0260	0.01575	0.00876	20.80	12.60	7.01
850	0.0262	0.01635	0.00904	22.27	13.90	7.68
900	0.0263	0.01695	0.00925	23.67	15.26	8.33
950	0.0264	0.01745	0.00961	25.08	16.58	9.13
1000	0.0266	0.01785mes	h Ch antdaS harr	na / CLEGO	17.85	9.90
		1	Kanpur	1		103

Ostwald-deWaele power law model was applied to analyze the flow behavior index of the synthesized biolubricant.

$$\tau = K \gamma^n \qquad \qquad \dots \qquad (3)$$

where K = consistency index

n = flow behaviour index

- The logarithms were taken on both sides of eq. (3) and log τ versus log γ plots were constructed for each temperature value.
- The intercept of the resulting straight line provided the magnitude of log K and the slope gave n.

- Both the consistency index and flow behavior index are significant fluid properties and are considered constant at a given temperature.
- The power law classifies the fluids into three different categories based on their flow behavior index: newtonian (n = 1), pseudoplastic (n < 1), and dilatant (n > 1).
- The shear stress values of the synthesized biolubricant were predicted using power law model (eq. 3) at shear rate variation of 100, 250, 500, 750, and 1000 per second and temperatures of 27, 60, and 90°C.

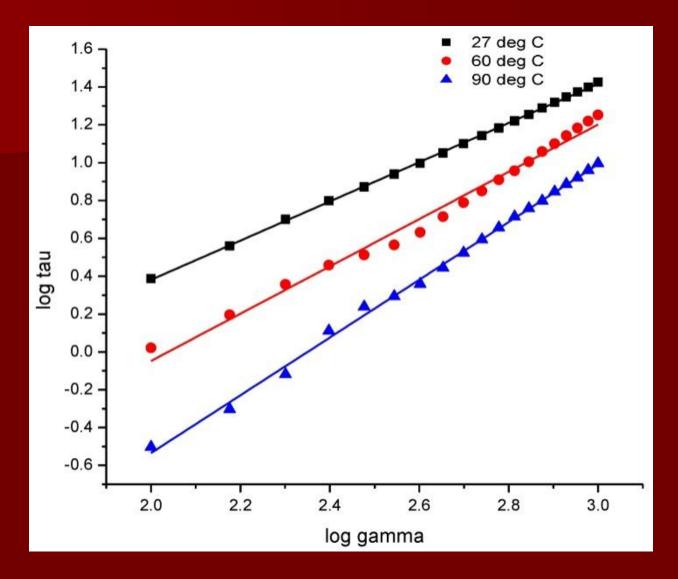


Figure: Power law behaviour of synthesized biolubricant at 27, 60, and 90° Gietemperatures Kanpur

Table 16: Power law parameters							
Temp (°C)	Temp (°C) n K R^2						
27	1.036	20.32×10^{-3}	0.999				
60	1.250	$2.83 imes 10^{-3}$	0.986				
90	1.527	$0.26 imes 10^{-3}$	0.997				

The flow behavior index (*n*) of the biolubricant sample ranged from 1.036 to 1.527, which indicated Newtonian fluid behavior at room temperature that gradually transformed to non-Newtonian behavior with increase in temperature.

The consistency index (*K*) for the sample was higher at lower temperatures. Little less than elevenfold increase in the magnitude of the K values between 90 and 60°C was observed, whereas more than sevenfold increase between 60 and 27°C was observed.

Table 17: Experimental and predicted (in parenthesis) shearstress values in Pa at different temperatures								
Shear rate (1/s)	$T = 27^{\circ}\mathrm{C}$	$T = 27^{\circ} \text{C} \qquad T = 60^{\circ} \text{C} \qquad T = 90^{\circ} \text{C}$						
100	2.44 (2.40)	1.05 (0.89)	0.31 (0.29)					
250	6.30 (6.20)	2.87 (2.81)	1.30 (1.19)					
500	12.60 (12.71)	6.14 (6.69)	3.34 (3.44)					
750	19.43 (19.34)	11.44 (11.11)	6.28 (6.39)					
1000	26.60 (26.06)	17.85 (15.91)	9.90 (9.91)					

Table 17 displays a good harmony between the experimental and the predicted shear stress values.

The power law model characterizes the biolubricant as near Newtonian at room temperature and non-Newtonian dilatant fluid at high temperatures. Dr. Umesh Chandra Sharma / UIET,

FRICTION BEHAVIOUR OF SYNTHESIZED BIOLUBRICANT

- The friction behaviour of synthesized biolubricant was analyzed using Ducom TR30L
 Four-ball wear test rig following standard test method of ASTM D 4172-94 A and B.
- The four-ball wear tester consists of three balls held stationary in a ball pot plus a fourth ball held in a rotating spindle.
- The balls were thoroughly cleaned with toluene before experiment.

- 10 ml sample was taken for each test.
- The test conditions were 15 and 40 kg load, operating temperature of 75°C, rotational speed of 1200 rpm and operation time of 1 hour.
- The wear produced on the three stationary balls is measured under a calibrated microscope and reported as the wear scar diameter (WSD).

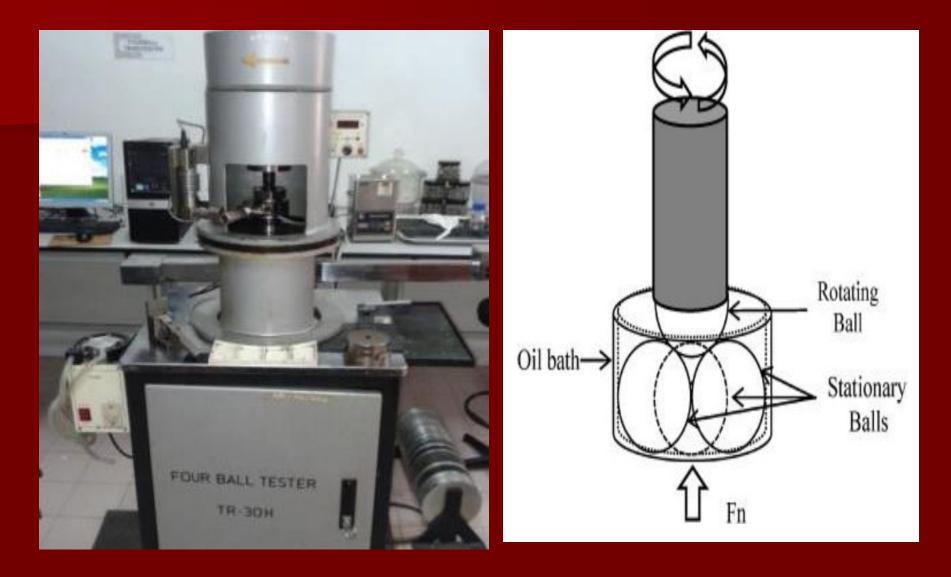


Table 18: Four-ball wear test results and comparison with reported data								
Sample	WSD mm (15 kg _f)	WSD mm (40 kg _f)						
КОТМРЕ	0.60	0.80						
POTMPE		0.78						
Ordinary lubricant		0.68						
Coconut oil		0.54						
SAE 20W50		0.36						

Aravind *et al.* (2015) mentioned that WSD values for vegetable oils range from 0.51-0.87 mm.

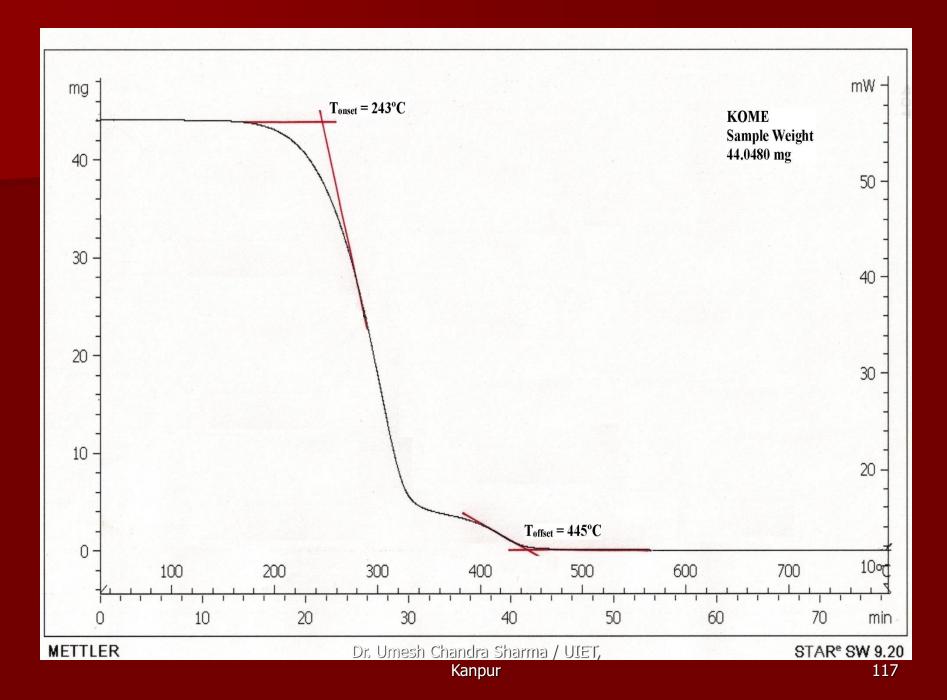
WSD values of 0.6 mm and 0.8 mm at the two loads of 15 kg and 40 kg were as expected based on knowledge from previous studies by other researchers.

The study was successful in establishing the application of synthesized biolubricant under high loads with satisfactory performance.

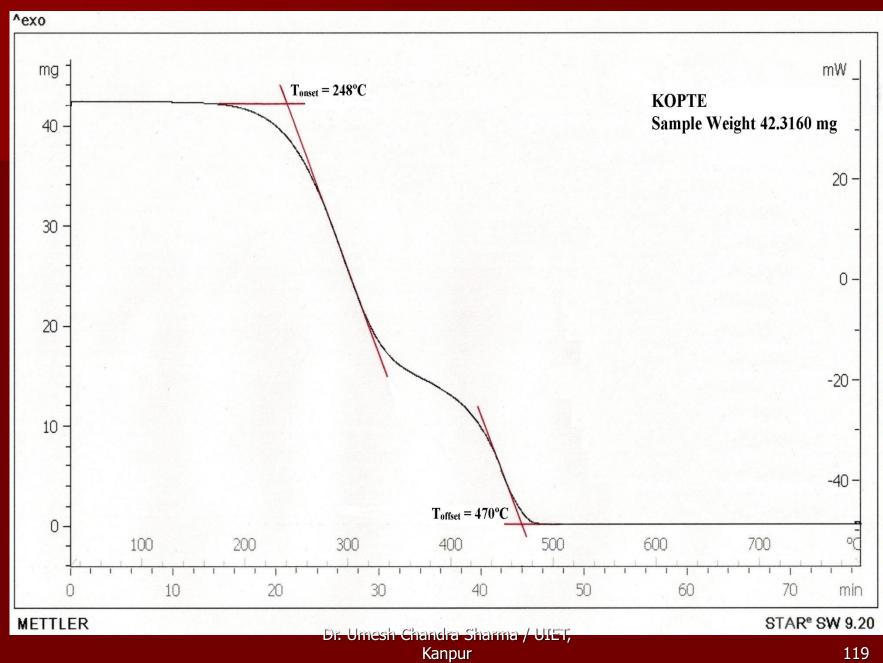
THERMO-OXIDATIVE STABILITY OF BIOLUBRICANT

- Thermal stability analyses of KOME and KOTMPE were performed in computer controlled Mettler
 Toledo thermogravimetric analyzer in non-isothermal mode under nitrogen atmosphere.
- Analysis was conducted at a heating rate of 10°C/min from 30 to 800°C temperature with nitrogen flow rate of 50 ml/min.
- TGA curves were used to analyze the onset and offset temperatures of the samples.

- The TGA degradation onset temperature indicated the resistance of the sample against thermal degradation and was determined by extrapolating the horizontal baseline of TGA curve at 1% degradation and the intercept of this line with the tangent to the downward portion of the curve.
- The higher onset temperature for product decomposition indicated the higher thermal stability.
- The maximum decomposition temperature approximated the temperature at which the maximum weight loss in the sample occurred.



- TGA data for KOME showed that 1% weight loss in the inert atmosphere occurred at 177.28°C.
- The 50% weight loss appeared at 289.36°C and 90% weight loss appeared at 340.83°C.
- The onset (T_{onset}) and offset decomposition (T_{offset}) temperatures were observed at 243°C and 445°C, respectively from the thermogram curve.
- The maximum decomposition temperature was observed at 302.63°C with 0.0868 mg weight loss.
- The temperature plateau starting at 469.17°C showed that no further significant weight loss was taking place.



- TGA data of KOTMPE showed that 1% weight loss occurred at 180.36°C.
- The 50% weight loss came off at 312.53°C and 90% weight loss took effect at 451.11°C.
- The onset and offset decomposition temperatures were observed at 248°C and 470°C, respectively.
- The maximum decomposition temperature appeared at 445.73°C with 0.07448 mg weight loss.
- The temperature plateau starting at 485.69°C showed that no further significant weight loss was taking place.

- Though the onset decomposition temperatures of KOME and KOTMPE were quite close, the maximum weight loss of KOTMPE at 445.73°C signified that KOTMPE was thermally more stable than KOME.
- Experimental data on thermal stability analysis of KOME and KOPTE along with data on thermal stability analyses of KOME, COME, COME epoxide, WCOME and WCOME epoxide as reported in literature are given in next Table 19.

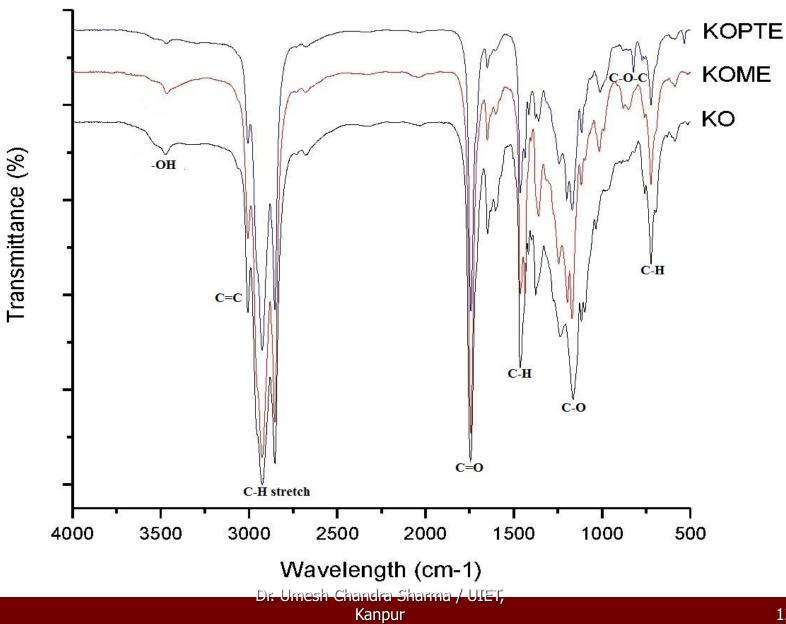
Table 19: Thermal stability analysis of KOME and						
KOTMPE						
Sample	T _{Onset} in nitrogen (°C)*					
KOME (Karanja)	243					
WCOME (Waste cooking oil)	175 – 178					
COME (Castor)	203					
KOME (Karanja)	205					
KOTMPE	248					
WCOME Epoxide	187					
Servo Hydraulic	260					
COME Epoxide	340					
* Heating rate 10°C/min.; # Gas flow rate 100 ml/min.						

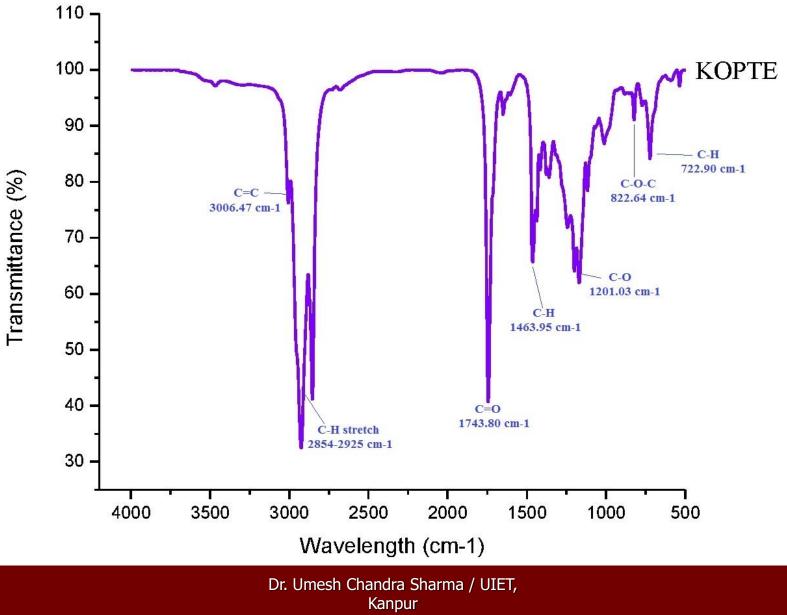
 ${\boldsymbol{\Box}}$

The weight loss profiles of KOME and KOTMPE in nitrogen atmosphere are given in Table 20.

Table 20: Weight loss profiles for KOME and KOTMPE								
Sample	Heating rate	Weight loss temperature (°C)						
	(°C/min)	1%	10%	50%	90%			
KOME	10	177	233	289	341			
КОРТЕ	10	180	241	312	451			

- Nik *et al.* suggested that the changes in specimen weight may be due to formation and disintegration of the physical and chemical bonds at elevated temperatures.
- From Table 20, It is evident that KOTMPE displays more thermal stability compared to KOME.
- There is marked difference in thermal stability of KOTMPE at 50% weight loss and onwards.
- This behaviour may be accredited to the removal of unsaturated bonds in fatty acid chains of KOTMPE, which leads to improved thermal stability of polyol triester.





KO	347	3.15	3006	6.64	2924.6	61 2854.12	1746.1	0	1649.	11	1604	.16	1464	4.75	14	17.88		
KOME	346	4.46	3006	5.20	2925.1	16 2854.42	1743.7	2	1650.	63			1464	4.07	14	36.09		
KOPTE	-		3006	5.47	2925.0	07 2854.46	1743.8	0	1650.	73			146	3.95	14	36.13	1	416.27
Functiona	-0)H	C=	C	C-H	stretching	C=O		C=C	1	C=C	С	C-	H	(C=C	C-	O-H in-
l Group	(alc	ohol	bend	ing	V	ibration	stretchin	ng			stretch	ning	sciss	orin	stre	etching	plan	e bending
-)	vibra	atio	(a	liphatic)	vibratio	n			U		g and		Ũ	(ca	rboxylic	
			n		, ,	• /	(ester)						bend					acid)
KO		137	7.46	123	8.44		1165.18	11	18.09	10	98.58	103	6.71			722.7	5	589.31
KOME	,	1360	0.94	124	6.20	1196.81	1170.76	11	18.40			101	7.15	852.	56	723.0	0	
KOPT	£	1360	0.28	124	3.52	1201.03	1169.72	11	16.84			101	1.30	822.	.64	722.9	0	535.23
Function	nal	C=	-0	C	-0	C-0	C-CO-			(C-0	C	-0	C-0	-C	C-H		
Group						stretching	С							stret	ch	grou	р	
					1	asymmetric										vibrati	on	
																(aliphat	tic)	

The presence of ester group in Polyol (TMP) triester was determined by the FTIR spectroscopy. KOME reacted with TMP using sulfuric acid as catalyst produced Polyol triester and water as byproduct.

- Based on the comparison of three spectrums of KO, KOME and KOTMPE, the peak of the hydroxy group (-OH) (3000-3500 cm⁻¹) did not appear in the spectrum of Polyol triester. This indicated that the esterification reaction was complete.
- The complete disappearance of C=C double bonds at wavelength 1604 cm⁻¹ in KO and appearance of C-O-C bands in esters (822 cm⁻¹) signified that most of the double bonds were converted.
- There was a wavelength that appeared after the esterification at 1196-1201 cm⁻¹ which showed the functional group of the C-O bond as the result of the formation of ester.
- The esterification rate calculated by acid values of the system before and after the reaction showed 87.35% conversion.

- The oxidation stability refers to the ability of the oil to maintain its properties, mainly its viscosity, when exposed to specific operating conditions.
- Triglycerides have low oxidation stability due to presence of unsaturated double bonds and their configuration.
- The exposure to heat, light, moisture and air could result in rapid oxidation leading to increased viscosity and polymerization.
- These changes in oil properties are irreversible and cannot be reversed by heating the oil.

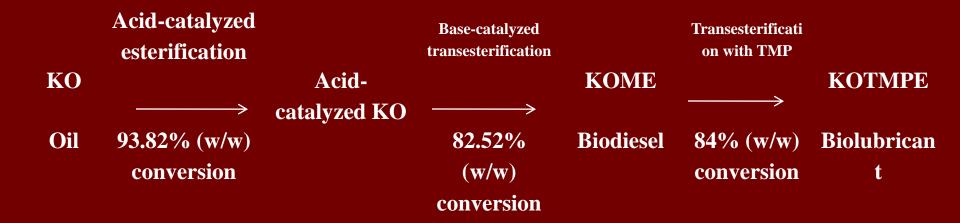
- Instead, an increase in oil temperature increases the rate of oxidation.
- Oxidation of oils and fats results primarily in the formation of hydroperoxides, which decompose into volatile aldehydes.
- Propyl gallate and tertiary butyl hydroquinone (TBHQ) may be added to increase oxidative stability, and t-butylhydroxy toluene (BHT) or t-butyl hydroxyanisole (BHA) may be added to increase shelf life.
- These additives may be used in amounts of up to 0.01% singly or 0.02% in combination.

- The oxidation stability of synthesized product was determined with Rancimat instrument according to EN 14112 accelerated oxidation test.
- The EN 14112 method expresses the oxidation stability of the tested material in terms of an induction period (OSI IP) for the production of volatile organic acids, which are by-products of fatty acid ester oxidative degradation with heat and oxygen.
- Oxidation Stability: 6 hours at 110°C

COST ANALYSIS AND POTENTIAL APPLICATIONS OF

SYNTHESIZED BIOLUBRICANT

General reaction scheme



Basis of calculation: 1 litre of KO

Step 1: Acid-catalyzed esterification of KO Density of KO at $27^{\circ}C = 0.9088 \text{ g/cm}^3$ Density of MeOH = 0.791 g/ml Density of H₂SO₄ = 1.835 g/ml Molecular weight of KO = 295.57 g/mol Molecular weight of methanol = 32.04 g/mol Mass of KO = 908.8 g

KO	H Methanol	+ H_2SO_4 catalyst	\rightarrow Acid-catalyzed
(908.80 g)	(591.14 g)	(9.09 g)	KO (852.64 g)
(3.075 mole)	(18.45 mole)		
(1000 ml)	(747.33 ml)	(4.95 ml)	

Step 2: Base-catalyzed transesterification

M.W. of acid-catalyzed KO = 347.87 g/mol

Acid-catalyzed	+ Methanol	+	KOH	\rightarrow	KOME	+	Glycerol
KO			catalyst				
(852.64 g)	(470.99 g)		(10.66 g)		(7 03.6 g)		(70.36 g)
(2.45 mole)	(14.7 mole)						
	(595.43 ml)						

Step 3: Transesterification of KOME to form KOTMPE

Molecular weight of KOME = 375.74 g/mol

KOME	+	TMP	+	H ₂ SO ₄ catalyst	\rightarrow	KOTMPE
(703.6 g)		(62.79 g)		(14.07 g)		(591.02 g)
(1.87 mole)		(0.468 mole)				
				(7.67 ml)		(641.85 ml)

Part A: Material cost

Table 21: Material cost calculation									
Material	Quantity u	ised	Unit price (Rs.)	Cost (Rs.)					
Karanja oil	1000	ml	53=00/litre	53=00					
Methanol	839	9.23	22=00/litre	18=46					
		ml							
Sulphuric acid	12.62	ml	12=00/litre	00=15					
Potassium	10.66	g	58=00/kg	00=62					
hydroxide									
Trimethylolpropa	62.79	g	135=00/kg	8=48					
ne									
Material cost				80=71					

Part B: Electricity cost

Table 22: Electricity cost calculation									
Process	Equipment used	Load (kW)	Duration (h)	Electricity consumed (kWh)					
Preheating of oil	Heater cum magnetic stirrer	0.418 kW	0.5	0.209					
Acid catalyzed esterification	Heater cum magnetic stirrer	0.418 kW	3.0	1.254					
Base catalyzed transesterification	Heater cum magnetic stirrer	0.418 kW	1.0	0.418					
Methyl ester transesterification	Heater cum magnetic stirrer	0.418 kW	3.5	1.463					
Methyl ester transesterification	Vacuum pump	0.187 kW	3.0	0.561					
Cooling water in reflux condenser	Water pump	0.018 kW	7.0	0.126					
Total electricity consumed	ET.	4.031							
Electricity cost @ Rs. 8=0		32=25 ¹³⁸							

Total cost (Rs.)=material cost (Part A) +
electricity cost (Part B)=80.71 + 32.25=112.96

From 1 litre of KO, 641.85 ml of KOTMPE biolubricant was synthesized. The calculated cost is Rs. 113. Cost for 1 litre of biolubricant will be Rs. 176. The process needs to be optimized before industrial production of this lubricant in order to maximize the profit.

Search for potential applications

- Following commercial lubricants were selected for the purpose of properties comparison study:
 - HP Milcy Turbo 15W-40 (Diesel Engine)
 - HP HDX Multigrade Engine Oil 20W-40 (Diesel LCV, HCV)
 - HP Gasenol Engine Oil 20W-50 (CNG, LPG and Dual Fuel Car, Taxi, Autorickshaw, Bus)
 - HP Racer 2 Two Stroke Engine Oil (Two Stroke Bike, Scooter, Autorickshaw)
 - HP Racer 4 Four Stroke Bike Engine Oil
 - HP Gear Drive EP 90
 - HP Kisan Shakti Pumpset Oil (Diesel Engine Pumpset)
 - HP Railroad oil 613
 - HP shock absorber oil
 - Castrol Activ Scooter 10W-30 (For Gearless Scooters)
 - Castrol GTX Engine^DOH 20 Wandra Sharma Peterol Cars)

Table 23: Comparison of key lubricating properties of synthesized and commercial lubricants

Product	Density (g/ml)	Viscosity @40°C (cSt)	Viscosity @100°C (cSt)	Viscosity index	Flash point (°C)	Pour point (°C)
Synthesized KOTMPE	0.9208 (27°C)	15.23	9.12	206.72	216 (min)	+ 6 (max)
HP Milcy Turbo 15W-40	NA	NA	14.0 - 15.0	120 (min)	214 (min)	– 24 (max)
HP HDX multigrade engine oil 20W-40	NA	NA	13.5 – 16.0	110 (min)	200 (min)	– 18 (max)
HP gasenol engine oil 20W-50	NA	NA	18.0 - 20.0	110 (min)	200 (min)	– 18 (max)
HP Racer 2 two stroke engine oil	NA	45 - 65	6.5	NA	70 (min)	– 15 (max)
HP Racer 10W-30 four stroke bike engine oil	NA	NA	10.8–11.8	120 (min)	196 (min)	– 27 (max)
HP Gear drive EP 90	NA	NA	16.5 – 18.0	90 (min)	180 (min)	- 9 (max)
HP Kisan shakti diesel pumpset oil	NA	NA	13.5 – 16.0	110 (min)	200 (min)	– 18 (max)
HP Railroad oil 613	NA	NA	15.8 – 16.2	90 (min)	225 (min)	– 6 (max)
HP shock absorber oil	NA	11.5 – 13.1	2.8 (min)	45 (min)	149 (min)	- 45 (max)
Castrol Activ scooter 10W- 30	0.87 (15°C)	76	11	144	196 (min)	– 27 (max)
Castrol GTX engine oil 20W-50	0.878 (15°C)	167	19	128	210 (min)	– 21 (max)
		Du Llussach Cha		TET		

- The density of synthesized biolubricant KOTMPE (0.9208 g/ml at 27°C) was higher than density of Castrol 10W-30 (0.87 g/ml at 15°C) and 20W-50 oils (0.878 g/ml at 15°C).
- The viscosity of KOTMPE at 40°C (15.23 cSt) was close to HP shock absorber oil (11.5-13.1 cSt) and at 100°C (9.12 cSt) was close to HP Racer 10W-30 four stroke bike engine oil (10.8-11.8 cSt) and Castrol Activ scooter 10W-30 oil (11 cSt).
- The viscosity of KOTMPE at 100°C was much better than HP shock absorber oil (2.8 cSt).
- The calculated viscosity index of KOTMPE biolubricant (206.72) was highest among all the lubricants compared here.
 Dr Umesh Chandra Sharma / UIET.

- The flash point of KOTMPE (216°C) was only little inferior to HP Railroad oil 613 (225°C) and was higher than rest of oils.
- The pour point of KOTMPE was greatly poor and needs to be taken care with addition of appropriate additive package.
- Here it was found that synthesized biolubricant KOTMPE is at par with most commercial lubricants in key lubricity properties and can be used for any given application with its pour point as the only cause of concern.

Thank you

0