

Fourier Transform Infrared Spectroscopy and the Physicochemical Properties of Modified *Plukenetia Conophora* and *Thevetia Peruviana* Seed Oils for Lubricant Purposes

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ABSTRACT:

Recently, there have been diversion of attention from lubricant from fossil sources to biolubricants due to its effect on the environment and human life. Vegetable oils have found usefulness in the production of biolubricant but have some drawbacks. In order to mitigate these problems, *Plukenetia conophora* (PKCO) and *Thevetia peruviana* (TVTO) seed oils were chemically modified via epoxidation, hydroxylation, and acetylation. Epoxidation was done using glacial acetic acid (0.05mol of acid: 1 mol C=C; Amberlite IR – 120H ion exchange resin; 1.5mol H₂O₂: 1 mol C=C) at 65°C for both PKCO and TVTO. The hydroxylation reactions were done at room temperature (97% formic acid; 30% H₂O₂) for 12hr. Acetylation from epoxidation was also carried out. Reactions were monitored by FTIR spectroscopy, and the physicochemical and lubricant properties investigated. The bands at 925-820cm⁻¹ confirms the presence of epoxy group in EPPKCO and EPTVTO. The band at 3450–3430cm⁻¹ indicate the hydroxyl group in both PKCO-OH and TVTO-OH. It also confirms hydroxyl acetal formation. EPPKCO has proven to be the best candidate for biolubricant formulation based on the physicochemical and lubricant properties, whereas the peroxide value must be improved using appropriate additives.

Keywords: Spectroscopy, Vegetable oils, Acetylation, Epoxidation, Hydroxylation.

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INTRODUCTION

Lubrication is a process that uses a chemical known as lubricant, to lower friction and wear between two surfaces that are in motion with each other. It helps in the transmission of pressure created between two opposing surfaces. Lubricants act as anti-friction agents [1]. In present day economy, sustainability has become an important factor, while on environment concerns list, resources and



energy conservation have also moved to the top. Due to the concerns on socioeconomic and environmental sustainability, lubricants emerging source of social awareness as commercial process is becoming more concerned about limitation of resources and commitment to generations to come. [2-4]. Lubricants are used in machines that are designed for industrial purposes. Proper functioning of machines and frequent failure reduction is aided using lubricants [5,6].

The demand for the replacement of lubricants from fossil fuel due to their depleting properties and price increase has become a global concern. Interest in the development of lubricants has led to an increased demand for biolubricants due to their features [7]. Biolubricants have advantages over fossil fuel, they are biodegradable, non-toxic, and environmentally friendly. With all the several advantages possessed by biolubricants, chemical modifications are still required to mitigate the issues of poor thermal and oxidative stability associated with it [8]. Conventional petroleum lubricants are frequently utilized in agricultural equipments, industrial machinery and automotives, but due to lack of strict regulatory measures, the excessive use of these products resulted to environmental pollution in relation to soil and water contamination [9]. Biolubricants are made from plants and animal fats, because of their outstanding record of qualities such as biodegradability, carbon neutrality to the environment [10] and the ecosystem remains unhurt during their spillage [11,12]. The carbon-neutral nature of biolubricant classified them as sustainable lubricants [13]. A biolubricant can be produced from non-edible (*Thevetia peruviana* (TVTO) and *Adenopus breviflorus*) or edible (*Plukenetia conophora* (PKCO), sunflower, olive, linseed oil, coconut, and rapeseed oil) sources [14,15]

Vegetable oils are example of biolubricant because of their inherent qualities, they are formed mainly by triglycerides which comprise three fatty acids that are associated with the glycerol backbone. The C=C in the structure of these triglycerides enable vegetable oil to be transformed [16,17]. There are two drawbacks associated with vegetable oils as lubricants: poor cold flow and poor thermo-oxidative stability properties [17,18]. Chemical modifications such as acetylation, epoxidation, hydroxylation, and transesterification have been carried out to improve on these inadequacies [19]. Many researchers have carried out epoxidation reaction of vegetable oils, alkyl esters and their unsaturated fatty acids to optimize the reaction and increase the yield of the epoxide. There are various methods that can be used in carrying out epoxidation: catalytic epoxidation using acid ion exchange resin [20], the conventional method used in industry [21], and chemoenzymatic epoxidation [22,23].

Epoxidized vegetable oils have been used as intermediates in the production of polymers which are commonly used as stabilizers, plasticizers, and reactive diluents [24-26]. Rubber seed oil was modified for the enhancement of its properties by Mohammed et al. [27]. Materials formulations by epoxidation of the rubber seed oil were carried out, opening of oxirane ring and transesterification led to the occurrence of acylation. A major decrease in the iodine value was observed after epoxidation. An overview of biolubricant forms, pathways development, properties and applications were reported by Cecilia et al. [28].

The aim of this present research is to study the Fourier transform infrared spectroscopy of epoxidized *Plukenetia conophora* (EPPKCO) and *Thevetia peruviana* (EPTVTO); hydroxylated *Plukenetia conophora* (PKCO-OH) and *Thevetia peruviana* (TVTO-OH); acetylated *Plukenetia conophora* (ACEPKCO) and *Thevetia peruviana* (ACETVTO) for the purpose of biolubricant formulations. The physicochemical and lubricant properties of these oils were also investigated.

MATERIALS AND METHODS

PKCO seeds were purchased from a local farmer in Iluomoba, Ekiti State, Nigeria. While the TVTO seeds were gotten from Federal Housing Estate, Ado-Ekiti. Bad seeds were removed, and the good seeds deshelled, washed with water, air-dried, and milled using electric blender into fine powder and kept in an air-tight bag until use. All the reagent used are of analytical grade.

Extraction of Oil Using Soxhlet Extraction Method

The oils of PKCO and TVTO were extracted in a Soxhlet apparatus with n-hexane, solvent was removed on a rotary evaporator. The oils were refined by agitating with 18M NaOH (1:30g/g) of alkali



powder for 15min. The resultant mixture was heated to 75 - 80°C for soap stock breaking, the neutral oil was separated by centrifugation.

Preparation of Epoxidized PKCO and TVTO Oil

TVTO (140g, 100mM C=C) and glacial acetic acid (30g, 0.05mols of acid; 1mol C = C) were placed in three-necked round bottom flask while stirring thoroughly at room temperature. 21.05g Amberlite IR-120H ion exchange resin was added, as the temperature was increased to 55°C, stirring continued for 30min. 170g H₂O₂ (1.5moles H₂O₂: 1C = C) was dropwisely added to the reaction mixture over 1 hr. Temperature was increased to 65°C on complete addition of H₂O₂ and reaction allowed to continue for 12hr. Rapid stirring was maintained throughout the experiments. The product mixture was poured into ice water and extracted with diethyl ether after the extraction. Dilute sodium bicarbonate was used to wash the ether layer, followed by distilled water until neutral to pH. The organic layer was dried over magnesium sulphate, and filtered under vacuum, removing the solvent over rotary evaporator [29]. Same procedure was used for preparing PKCO using correctly calculated amount of materials.

Preparation of Hydroxylated PKCO and TVTO

100ml of PKCO was added to 100ml of 97% formic acid in a three- necked flask coupled with thermometer and mechanical stirrer. The mixture was placed and stirred in an ice-bath between 10 – 15 °C. 85 ml of 170 ml of 30% H₂O₂ for PKCO while keeping the temperature still at 10 – 15 °C. The reaction was allowed to continue at room temperature for 12hr on the complete addition of the H₂O₂. Diethyl ether was used for extraction of the emulsion. The ether layer was washed respectively with water, saturated sodium hydrogen carbonate followed by saturated sodium chloride. The ether layer was dried over sodium sulphate (water free) and solvent removed over rotary evaporator [30]. This was repeated for TVTO.

Preparation of Acetylated PKCO and TVTO

50g of hydroxylated PKCO was added to pyridine (16ml, 0.19mol), and acetic anhydride (0.19mol). The mixture was stirred at 80°C for 3 h. Excess acid anhydride was destroyed by the addition of Water (30mL) and pyridine (10mL) and stirred at 60°C for 2 hr. The reaction mixture was cooled to room temperature and diluted with 100mL ethyl acetate, washed with water (100mL), 10% NaOH (2x 100mL), 10% HCl (100mL) and 5% aqueous sodium bicarbonate successively. The reactant was dried over anhydrous MgSO₄ and evaporated to give the product.

Spectroscopic Analysis

The Fourier Transform Infrared (FTIR) analysis would be carried out to determine the functional group of the PKC and TP. The spectra would be recorded on an FTIR Perkin-Elmer spectrophotometer. A potassium bromide (KBr) pellet would be used to determine the background signal. The spectral would then be obtained over the frequency range 4000-650 cm⁻¹ at the resolution of 4cm⁻¹ and the final output would be in % transmittance [31].

Physicochemical Properties

Properties like iodine (IV), peroxide (PV), and saponification (SV) values for each of the samples were obtained in triplicate based on the standard methods of the American Oil Chemist's Society [32-25]. viscosity at 40 and 100 °C was measured as a function of shear rate, which was varied from 100 to 1 s⁻¹, as described by Biresaw and Bantchev, [36].

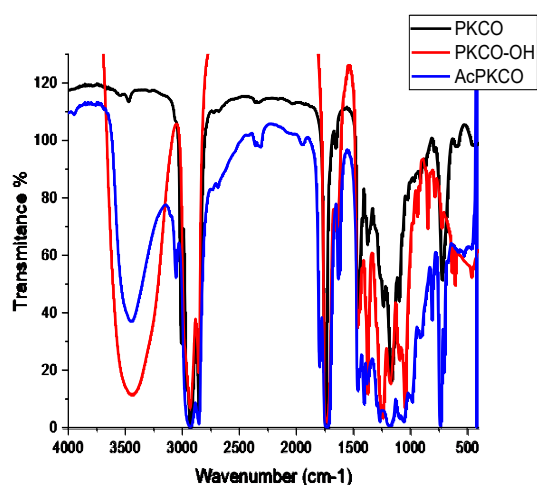
RESULTS

FTIR Spectroscopy Result of PKCO and TVTO

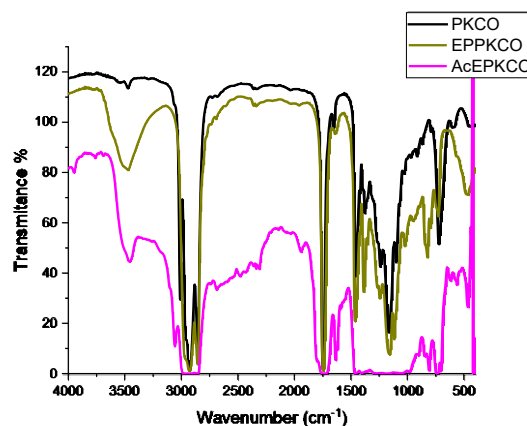
The absorption peaks were monitored by FTIR spectroscopy to determine the functional groups of PKCO and TVTO in comparison with their chemically modified counterparts. The FTIR spectra of PKCO, PKCO-OH, EPPKCO, ACEPKCO and TVTO, TVTO-OH, ACETVTO, and EPTVTO are represented in Figures 1a, b and 2a, b respectively. The following are the main regions shown by all the spectra: C-H (methane hydrogen atom) at 2920 - 2815 cm⁻¹, C-H (alkene vibration) at 750 - 718



cm^{-1} , C=O stretching at $1750 - 1745 \text{ cm}^{-1}$, -C-O stretching at $1200-1100\text{cm}^{-1}$ and O-H stretching vibration at $3450-3430\text{cm}^{-1}$.

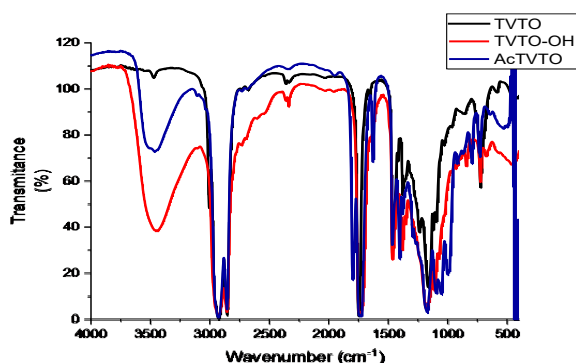


(a)

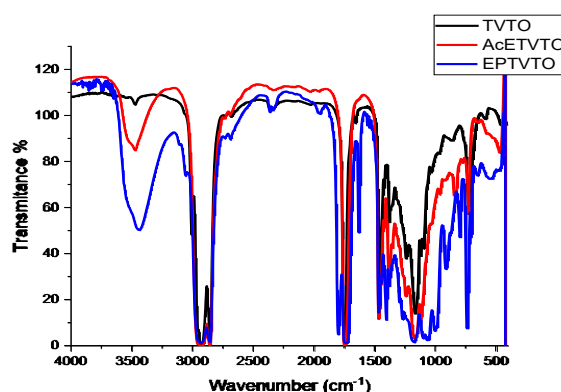


(b)

Figure 1. FTIR of (a) PKCO, PKCO-OH and AcPKCO (b) PKCO, EPPKCO, AND AcEPPKCO



(a)



(b)

Figure 2. (a) FTIR of TVTO with (a) TVTO-OH and ACTVTO; (b) ACETVTO and EPTVTO

Table 1 and 2 show the results of the physicochemical and lubricant properties of chemically modified PKCO and TVTO seed oils respectively. However, the highest, and lowest saponification values (364.65 mg KOH/g and $78.54 \text{ mg KOH/g oil}$) were observed in ACEPKCO and EPPKCO respectively. While the highest iodine value ($23.85 \text{ gI}_2/100\text{g}$) and peroxide value (120 Meq KOH/g) were observed in TVTO. At temperatures 40°C and 100°C , both ACEPKCO and PKCO-OH had an outrageous higher value $5,700\text{cSt}$, $8,639\text{cSt}$ and 169.24cSt , 225.72cSt respectively compared to others. Conversely, there was a significant increase in viscosity index of TVTO (238) as the temperature increased. Although, both epoxidized and hydroxylated TVTO had the highest cloud point (27°C) with PKCO having the lowest value (-9°C).



Table 1. Physicochemical Properties of Chemically Modified *Plukenetia conophora* and *Thevetia peruviana*

Sample(s)	Saponification mg KOH/g oil	Iodine (gl ₂ /100g)	value	Peroxide (Meq KOH/g)	value	Colour
PKCO	173.91	16.75		38.00		1.50
EPPKCO	78.54	1.58		50.00		0.00
PKCO-OH	185.13	2.33		0.00		0.50
ACEPKCO	364.65	2.50		0.00		3.00
TVTO	173.91	23.85		120.00		0.00
EPTVTO	196.35	3.81		36.0		0
TVTO-OH	252.45	2.03		0.00		0
ACETVTO	280.50	2.50		0.00		0.50

Table 2. Lubricant properties of chemically modified *Plukenetia conophora* and *Thevetia peruviana* seed oils

Sample(s)	Viscosity @40 (cSt)	Viscosity @ 100 (cSt)	Viscosity index	Cloud point (°C)	Pour point (°C)
PKCO	8.36	2.50	131	-9	-24
EPPKCO	162.90	23.15	171	-6	-9
PKCO-OH	8,639	225.72	116	39	-
ACEPKCO	5,700	169.24	111	-	-3
TVTO	25.68	6.69	238	12	-3
EPTVTO	87.02	15.94	197	27	3
TVTO-OH	354.88	45.91	189	27	-12
ACETVTO	327.82	34.58	150	-3	-18

DISCUSSION

The O-H peak at 3450cm⁻¹ observed in EPPKCO and EPTVTO was due to the emergence of epoxides in the presence of H₂O₂ [37]. The small peak observed in PKCO and TVTO could be because of the terminal hydroxyl groups in their glycerol backbone chain [38]. The characteristic peaks that are significant of C-O-C stretching from oxirane vibrations at 925-820cm⁻¹ and 1100cm⁻¹ are observed in the FTIR spectral of both EPPKCO and EPTVTO [1,39]. The non-appearance of any band around 3000-3450cm⁻¹ is an indication that during epoxidation process, no oxirane cleavage took place [40]. The appearance of the strong multiple bands at 1000- 1180cm⁻¹ in ACETVTO and ACEPKCO indicate O-C-O. The emergence of O-H peak (broad band) at 3450cm⁻¹ in the spectra of acetylated PKCO and TVTO further confirms the hydroxyl acetal formation. An additional confirmation of acetal formation is the broadening of carbonyl (C=O) peak at 1750cm⁻¹ [41,42].

The result shows that ACEPKCO has a higher saponification value than the value recommended by WHO. High saponification value depicts a high triacylglycerol content which is an indication that the oil would be useful in cosmetic industries [43] and not for lubricant as expected since high saponification value is a pointer to increased ester bonding, which indicate a lower hydrolytic stability. While the lowest saponification value exhibited by EPPKCO makes it relevant for lubricant formulation.

Similarly, EPPKCO and TVTO-OH had the least iodine values 1.58 and 2.03gl₂/100g respectively. This can be attributed to reduced degree of unsaturation in both oils due to structural re-orientation caused by both epoxidation and hydroxylation, it can be concluded that each of the double bond has been replaced by one epoxyl and hydroxyl group [44]. The IV is used to measure the degree of unsaturation per 100 g of sample, and according to the IV, the Vegetable oils can be grouped as drying oils (IV > 130), semi-drying oils (90 < IV < 130), and non-drying oils (IV < 90) [45,46]. Both PKCO and TVTO with iodine value 16.75 and 23.85 gl₂/100g respectively are polyunsaturated oil which are desired by oil processors [43]. This observation shows that the degree of unsaturation in



EPPKCO has been reduced and this may have impacted on its better oxidative storage stability and makes it fit for lubricant purposes. The peroxide value gives a measure of the extent to which an oil sample has undergone oxidation (rancidity). It is important to note that higher peroxide values observed in TVTO (120 Meq KOH/g), EPTVTO (36 Meq KOH/g), PKCO (38 Meq KOH/g) and EPPKCO (50 Meq KOH/g) may affect their storage time. This may be because of the reaction of H_2O_2 in EPPKCO and EPTVTO while the value observed in TVTO and PKCO may be due to storage conditions as oils exposed to both air and light during storage showed increase in peroxide value [47].

The increase in the use of biodegradable products has been driven by the high environmental hazard attributed to the use of lubricants from mineral or fossil sources. Vegetable oils are becoming potential replacement for mineral-based oils due to their environmental friendliness, renewability, high lubricating properties such as good kinematic viscosity, excellent viscosity index, cloud point and pour point [48,49]. In this study, the kinematic viscosities at 40 and 100°C are extremely high in PKCO-OH (8639 and 225.72cSt) and AcEPPKCO (5,700 and 169.24cSt) respectively. This means that the two oils are too viscous and could only be suggested as solid biolubricant in partial replacement of those formulated from mineral sources, as plant oils are biodegradable in nature, and a renewable resource that reduces dependence on petroleum oil [50]. PKCO and TVTO had the poorest viscosities 8.36 and 25.68cSt respectively. This is an indication that both oils cannot be used as lubricant in their natural state unless they are chemically modified, as a good lubricant must possess better viscosity. Both the kinematic viscosities at 40 and 100°C (162.90 and 23.15cSt) with the viscosity index (171) are perfect for EPPKCO compared to others. This makes EPPKCO a potential alternative to the use of lubricant from fossil fuels among all the samples analysed.

CONCLUSION

Chemical modifications of PKCO and TVTO seed oils were carried out via epoxidation, hydroxylation, and acetylation. The reaction was monitored by FTIR spectroscopy, and their physicochemical and lubricant properties were investigated. The peaks at $925-820cm^{-1}$ confirmed the complete conversion of the C=C bond to epoxyl group in EPPKCO and EPTVTO. The hydroxyl groups were indicated in PKCO-OH and TVTO-OH by the broad band at $3450-3430cm^{-1}$. The hydroxyl acetal formation was also established. Observations from the physicochemical and lubricant properties project EPPKCO as the best among all the modified samples that can serve as a potential alternative to the use of lubricants from fossil sources if treated with appropriate additive to improve on its peroxide value.

CONFLICT OF INTERESTS

No conflict of interest.

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