JASEM ISSN 1119-8362

Full-text Available Online at
https://www.ajol.info/index.php/jasem
http://ww.bioline.org.br/ja

J. Appl. Sci. Environ. Manage.
Vol. 22 (5) 797 – 802 May 2018

Fatty Acid Methyl Ester of Nigerian Spent Palm and Peanut Oils: Non-Food Option for Biodiesel to Safe Food Security and Environment (Part I)

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ABSTRACT: In the awakening of Nigeria biofuel economy and reduced carbon footprint revolution, her targeted feedstocks such as sweet sorghum and palm oil are hidden threat to food security. To avoid this scenario, the present study derived fatty acid methyl esters (FAME) from Nigerian spent palm and peanut oils (NSPO and NSPeO) as cheap and non-food feedstocks for biodiesel and safe environment. Fresh and spent Nigerian palm oil (NPO) and peanut oil (NP_eO) were converted into FAME by one and two steps alkali transesterification using 6:1 molar ratio of methanol to oil, 1.0 % wt. potassium hydroxide pellets as catalyst at 60ºC over 1 hr. Characterization of feedstocks and FAME were carried out using European (EN) and USA (ASTM) norms for quality biodiesel. The quality of final FAME obtained after two-steps alkali transesterification were within international norms for biodiesel except for alkali and alkaline earth metals that required further removal using adsorption process as a post-transesterification treatment. The present study reveals Nigerian spent palm and peanut oils as potential non-food feedstocks for biodiesel production to safe food security and environment.

DOI: https://dx.doi.org/10.4314/jasem.v22i5.35

Dates: 09 January, 2018, Revised: 02 May 2018, Accepted: 05 May 2018

Key words: Alkali-transesterification, fatty acid methyl ester, food security, Nigerian spent palm

Nigeria needs to prepare for post-fossil fuel future to achieve a buoyant and sustainable economy. The insecure fuel supply, global warming, high environmental pollution and needs for agricultural products diversification are thundering a race to produce fuel from renewable resources in Nigeria. At her recent sensitisation workshop on biofuels development with the theme, ‘Biofuels: Nigeria’s New Economy’, Nigerian Minister of State for Petroleum Resources, said “Time has come for us to explore alternative energy and revenue sources with the abundance of land and great vegetation available at our disposal” (Biofuel International, 2017). Biodiesel is increasingly being used commercially for transportation to reduce GHG emission, decrease dependence on fossil fuel and support agriculture (World Bank, 2007). Biodiesel is reported to have similar combustion properties as diesel fuel with higher viscosity and octane number and thus considered a clean fuel and rated as a strong alternative candidate for conventional diesel fuel. Other advantages of biodiesel over conventional diesel include its environmental friendliness due to generation of lesser total unburned hydrocarbons and carbon dioxide. Also, pure biodiesel is non-toxic, biodegradable, with lower mutagenicity and therefore, could cause good reduction in cancer risk (Gilman *et al*., 2014; Tesfa *et al*., 2014).Nigeria proposed feedstocks for her first generation biodiesels are sugarcane, cassava, sweet sorghum, palm oil, and jatropha. However, some Nigerian chosen biodiesel feedstocks such as palm oil and sweet sorghum have hidden potential to trigger food insecurity in a country such as Nigeria having high percentage of hungry people, recent economy recession, and growing population with increasing demand for food and energy security. Nevertheless, the Estelvina *et al*. (2011) analysis of the effect of biodiesel on food safety indicates trend of rising food prices with increasing oil prices but not increasing production of biofuel directly due to its insignificant proportion in the world energy supply. Also, they emphasized the indispensable production of biodiesel due to its sustainability as energy is still much food source-dependent. Whatsoever, second generation renewable resources for biofuel are necessary for sustainable development to meet the essential needs of the present generation without compromising the ability to meet the essential needs of the future generations. Waste vegetable oils WVOs are oils that have altered physicochemical properties due to use in batch or continuous processing in food preparations (Gilman *et al*., 2014). Spent vegetable oils (SpVOs) are type of WVOs that are no longer useful for recycling into food preparations for

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they are no longer healthy and safe for consumption due to high carbon residues and oxidation products that are toxic to human body (Felizardo *et al*., 2006). The lack of information by the manufacturers makes the consumers directly discard SpVOs in sinks, toilets or even in the rivers and soil, contributing environmental impacts. WVOs have been successful used for production of biodiesel in USA, Canada, China, Austria and Germany (Barabás and Todorut, 2011; SENER, 2017). Nonetheless, SpVOs are promising feedstock for biodiesel production without competition or threat to food security, thereby relieving the burden placed on edible oils and food crops for biofuel production. Proportion of biodiesel generating from SpVOs, though, may not be sufficient but could complement biodiesel as blending agent to lower reliance on food crops and overall cost. In addition, diversifying SpVOs into biofuel production has a potential in reducing environmental impacts of GHG and CO_2 emission and thereby translating to safe and healthy environment (Felizardo *et al.,* 2006; Bart *et al*., 2010). Furthermore, WVOs and SpVOs meet some of the requirement for economic exploitation as cheap raw materials with low cost process (Canakci, 2007; USFDA, 2017). Therefore, as part of concerted efforts in search of complementing non-food bio-resources for biodiesel production in Nigeria, the present study assessed the potential of SpVOs as bio-resource for FAME production as standard biodiesel using one and two steps transesterification process.

MATERIALS AND METHODS

All experimental analyses were carried out in the Laboratory of Department of Chemical Sciences, Tai Solarin University of Education, Ijebu-Ode, Nigeria.

Vegetable oils sampling: Vegetable oil feedstocks for FAME production were locally obtained in National Bodija market, Oyo state, Nigeria. Fresh and unused Nigerian palm oil (NPO) and peanut oil (NP_eO) were purchased while Nigerian spent peanut oil (NSPeO) and palm oil (NSPO) were obtained freely from traditional bean cake sellers and restaurants after countless number of reuse for frying and reserved for disposal. Three samples were collected randomly per sub sections within three areas and pooled together per area as replicates, making three replicates for all experiments.

Methanolysis by One and Two steps alkali-transesterification: Clean and unused NPO and NPeO samples were used directly for transesterification process while NSPO and NSPeO samples were heated and filtered to remove any residues from processing and storage and thereafter, washed with warm water by gentle mixing to remove added-spices and left overnight in separating funnel and the water was run off and top-layer clean oil collected. One and two steps alkali-methanolysis for conversion of feedstock into FAME followed Bakir and Fadhil (2011) with modifications. In the one step transesterification, the feedstock were converted into FAME using 6:1 molar ratio of methanol to oil, 1.0 % wt. potassium hydroxide (0.025M KOH) pellets as catalyst and transesterification is carried out at 60°C over 1 hr, considering high AV of the feedstock. The biodiesel filtrate was washed consecutively with dil. H_2O until neutral to remove residual by-products such as excess alcohol, catalysts, soap and glycerine and then vacuum dried. In two steps alkali-transesterification, biodiesel obtained in the first transesterification was subjected to a second alkali-transesterification under same condition for one step-transesterification.

Characterization of feedstock and FAME: Availability of tools, cost, safety, disposal and health issues were considered for analysis of biofuel characteristics of feedstocks and FAMEs. Specific gravity (SG) was measured using gravimetric method. Density at 15°C (g/cm^3) was by densimeter according to EN-ISO 3675. The Kinematic viscosity at 40°C (mm^2/s) was by Ferranti portable viscometer measurement according to EN 3104. Flash point (FP, °C) followed D 93 and total water and sediment was by centrifugation as described in D 2709. Sulphated ash (ash, % mass) followed D 874. Conradson carbon residue (CC, % mass) after evaporation and pyrolysis was according to D 189, 100% distribution was estimated for crude feedstocks and 10% for distilled sample for FAMEs. Total acid value (AV, mg KOH/g) was estimated using macro method of titration after Pr EN 14104. Alkali metals were estimated using the sulphated ash according to EN 14108 and EN 14109 for Na and K, respectively and alkaline metals and heavy metals followed AOAC, 2005 using Atomic Absorption Spectrometer (AAS, Buck Scientific, USA).

Data Management: All data analyses for descriptive, ANOVA, and correlation were carried out using SAS version 9.2 (SAS, 2002).

RESULTS AND DISCUSSION

Characteristics of NPO, NPeO, NSPO, and NSPeO as biodiesel: Table 1 presents descriptive statistics for the biofuel properties of the assessed feedstocks and reveals higher mean viscosity, AV, FP, CC, ash, water and sediment for all the feedstocks when compared to standard norms for biodiesel (ASTM D6751, EN 14213 and EN 14214). This is no news, for there are several reports on the deficiency of SVOs as direct biofuel in diesel engines with regards to their very high viscosity, due to high molecular weight of their fatty acid composition, which increases with increasing carbon chain length and degree of saturation (Altin *et al*., 2001; Giakoumis, 2013). Higher viscosity, AV, and CC were observed for SpVOs over neat SVOs, which might be due to heterogeneous mixture of the former constituting particulate matter, organic impurities, oxidative degradation and polymerization products (Bart *et al*., 2010). The very high viscosity of the assessed feedstocks could cause ignition delay due to low volatility and poor fuel atomization, and therefore, they are not suitable in modern direct-injector diesel fuel system and engines. Their high FP contributes low volatility not suitable for cold start, and high AV and CC will result into carbonisation of injector strips, ring sticking, lubricating oil dilution and degradation. High biofuel characteristic of VOs as presently observed and their aforementioned defects resulted into mechanical damage and engine failure that led to their abandon as straight biofuel (Bart *et al.*, 2010]. Figure 1 shows group I and II metals exceeded values for biodiesel (EN 14214; ASTM D 6751) with exception of NPeME for only group II. Presence of these metals could emanate from diet, manufacturing and food processing of Nigerians and could cause corrosion of combustion chambers (Haseeb *et al*., 2010).

Table 1: Fuel properties and elemental content of Nigerian vegetable oil feedstock for FAME production

Fuel properties	Mean	SD	SE	CV	Range	Min	Max	N	Standard
NPO									EN 14214
Density kg/L	0.912	0.001	0.001	0.11	0.002	0.911	0.913	3	0.86-0.9
Viscosity mm^2/s	39.943	0.218	0.126	0.545	0.42	39.7	40.12	3	3.5 – 5.0
SG	0.887	0.006	0.003	0.651	0.01	0.88	0.89	3	
AV mg/KOH/g	5.863	0.257	0.148	4.386	0.48	5.57	6.05	3	0.5
FP	175.33	1.155	0.667	0.659	2	174	176	3	120
CC % mass	0.14	0.02	0.013	1.44	0.04	0.12	0.16	3	0.3
Ash % m/m	0.22	0.01	0.006	4.545	0.02	0.21	0.23	3	0.02
H_2O & sed	0.071	0.004	0.003	0.47	0.016	0.061	0.077	3	
NPeO									ASTM D6751
Density kg/L	0.896	0.002	0.001	0.171	0.003	0.894	0.897	3	
Viscosity mm^2/s	39.1	0.361	0.208	0.922	0.7	38.7	39.4	3	1.9-6.0
SG	0.873	0.006	0.003	0.661	0.01	0.87	0.88	3	
AV mg/KOH/g	0.977	0.133	0.077	13.635	0.24	0.89	1.13	3	0.05
FP	175.667	0.577	0.333	0.329	1	175	176	3	130
CC mass	0.076	0.003	0.002	0.38	0.005	0.074	0.079	3	0.05
Ash % mass	0.107	0.015	0.009	14.321	0.03	0.09	0.12	3	0.02
H_2O &sed	0.04	0.001	0.001	0.21	0.006	0.036	0.042	3	
NSPO									
Density g/cm^3	0.934	0.003	0.002	0.321	0.006	0.931	0.937	3	
Viscosity mm^2/s	55.413	0.707	0.408	1.276	1.32	54.9	56.22	3	
SG	0.9	0.01	0.006	1.111	0.02	0.89	0.91	3	
AV mg/KOH/g	9	0.396	0.229	4.401	0.78	8.57	9.35	3	
FP	193.667	1.528	0.882	0.789	3	192	195	3	
CC % mass	0.31	0.023	0.013	0.73	0.05	0.28	0.33	3	
Ash % m/m	0.347	0.023	0.013	6.662	0.04	0.32	0.36	3	
H_2O & sed	0.32	0.024	0.013	0.72	0.05	0.27	0.32	3	
NSPeO									
Density g/cm^3	0.939	0.002	0.001	0.213	0.004	0.937	0.941	3	
Viscosity mm^2/s	51.403	0.698	0.403	1.357	1.35	50.83	52.18	3	
SG	0.907	0.006	0.003	0.637	0.01	0.9	0.91	3	
AV mg/KOH/g	9.317	0.168	0.097	1.804	0.33	9.17	9.5	3	
FP	188.667	0.577	0.333	0.306	1	188	189	3	
CC % mass	0.35	0.03	0.02	0.88	0.06	0.32	0.38	3	
Ash % mass	0.367	0.015	0.009	4.166	0.03	0.35	0.38	3	
H_2O & sed	0.37	0.03	0.03	0.91	0.08	0.034	0.042	3	

SD, standard deviation, SD, standard error, CV, coefficient of variation, NPO, Nigerian palm oil, NPeO, Nigerian peanut oil, NSPO, Nigerian spent palm oil, NSPeO, Nigerian spent peanut oil, SG, specific gravity, AV, acid value, FP, flash point, Ash, ash content.

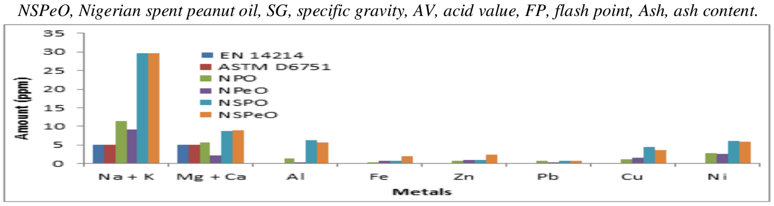

Fig 1: Comparative level of metallic content of NPO, NPeO, NSPO, NSPeO with EN 14214 and ASTM D 6751 standard. *NPO, Nigerian palm oil; NPeO, Nigerian peanut oil; NSPO, Nigerian spent palm oil, NSPeO, Nigerian spent peanut oil.*

One step transesterification of NPO, NPeO, NSPO, and NSPeO into FAME: biofuel characteristics of NPO, NPeO, NSPO, and NSPeO were improved by conversion into FAME using one step alkali-methanolysis. Based on Fig 2a, FAME from neat SVOs, NPME and NPeME, were within specified biofuel standard except for higher AV and CC for NPME while FAME of SpVOs, NSPME and NSPeME exceeded limits. The observed difference in the biofuel properties between FAME from neat and spent vegetable oils could be due to high, AV, FFA and water content characterized of FAME from frying oils (Felizardo *et al*., 2006; Canakci, 2007; Gashaw and Teshita, 2014). Also, according to Fig 2 (b), the FAME' yield obtained from neat SVOs were higher than those of SpVOs and generally, the present yields were lower to those reported in many literatures (Felizardo *et al.,* 2006; Canakci, 2007; Diya'udeen *et al*., 2012; Filho *et al*., 2014). The lower yields obtained at present are indication of incomplete transesterification of glycerides and fatty acids content of the feedstock, which deactivated the catalyst and with formation of soap during transesterification process (Meher *et al*., 2006). It worth mentioning that the levels of the metals were reduced (data not shown) which might be due to losses of some inorganic salt during washing and neutralisation steps but were not within specified values for group I and II metals. Presence of group I and II metals in fuel could lead to corrosion of the combustion chambers, engine knocking and many mechanical issues cause by high temperature, and their presence might be from adsorption by parent plant from soil, incorporation during oil and food processing or alkali residues in transesterification process (Haseeb *et al*., 2010) Therefore, one step transesterification caused improved biofuel properties of the SVO on conversion into FAME but biodiesel standards were not met in all FAME produced. In addition, the present yields of <80% are commercially undesirable, compared to >96.0% recommended by EN 14214. Many previous reports reveal the advantages of two steps involving acid-esterification for increasing FAME yields and improved biofuel properties for conversion of SVOs, WVOs and SpVOs into biodiesel (Filho *et al*., 2014; Diya'udeen *et al*., 2012; Canakci, 2007; Meher *et al*., 2006). However, the more recent two steps alkali-transesterification, that is akali-alkali transesterification reported by Bakir and Fadhil (2011) showed improved yield and biofuel characteristics over acid-base transesterification and therefore, was employed in further experiment.

Two-steps alkali transesterification of NPO, NPeO, NSPO, and NSPeO into FAME: The present two steps alkali transesterification similar to Bakir and Fadhil (2011) was able to derive FAME within EN 14214 and ASTM D6751 standards but still with higher AV and CC for NSPME and NSPeME (Fig. 3a). Generally, FAMEs produced possess higher FP. However, FAME with a yield >80% (v/v) was finally obtained (Fig. 3b), which corroborates yields in many similar studies (Felizardo, 2006; Canakci, 2007; Diya'udeen *et al*., 2012; Filho, *et al*., 2014). The present mean yields of FAMEs were higher than mean yields of fatty acid ethyl esters (FAEE) obtained from used vegetable oils using silica gel adsorption as post acid-base transesterification treatment by same research team (In press), corroborating Bakir and Fadhil (2011). Figure 4 shows combined alkali metals (Na + K) was still not generally desirable in all the obtained FAME and alkaline earth metals (Mg + Ca) also exceeded limit in NSPME and NSPeME (5 ppm in EN 14214; ASTM D6751. Therefore, none of the present FAME produced with two steps alkali-transesterification is a biodiesel according to European norm (EN 14214) considering their metallic content.

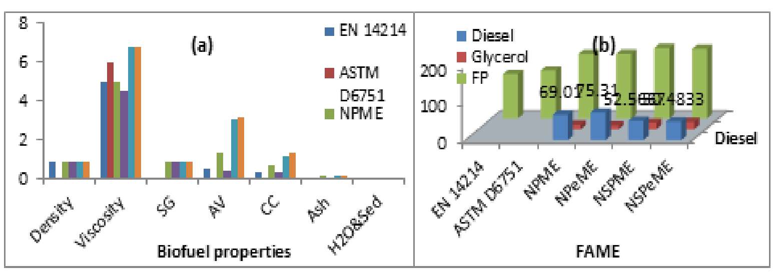

Fig 2: Biofuel properties of derived FAME, NPME, NPeME, NSPME, and NSPeME (a) and Yield of biodiesel, glycerol and FP of FAME (b) in one step alkali-transesterification in comparison with European and USA biodiesel standards. *NPME, Nigerian palm methyl ester; NPeME, Nigerian peanut methyl ester; NSPME, Nigerian spent palm methyl ester; NSPeME, Nigerian spent peanut methyl ester; SG, specific gravity; AV, acid value; CC, Conradson carbon residue, ash, sulphated ash; H2O&Sed, water and sediment.*

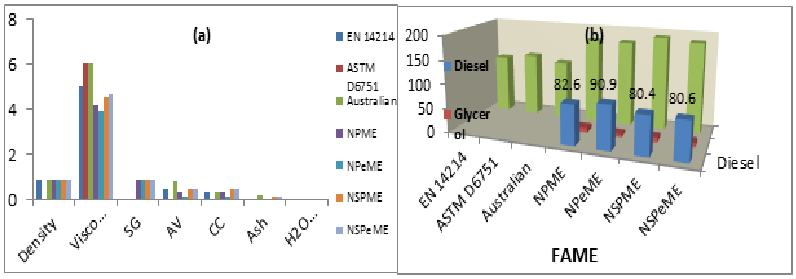

Fig 3: Biofuel properties (a) and Yield of biodiesel, glycerol and FP (b) of NPME, NPeME, NSPME, and NSPeME after two steps alkali transesterification. *NPME, Nigerian palm methyl ester, NPeME, Nigerian peanut methyl ester, NSPME, Nigerian spent palm methyl ester, and NSPeME, Nigerian spent peanut methyl ester.*

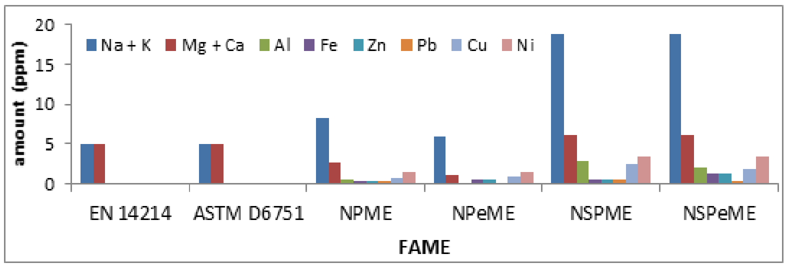

Fig 4: Level of metals in NPME, NPeME, NSPME, NSPeME after two steps transesterification.

Table 2: Pearson Correlations for useful relationships among oil feedstock and Biofuel properties

	Density	Viscosity	SG	AV	FP	CC	Diesel	Glycerol	Ash	Na	K	Mg
Freshness	0.33*	ns	0.65***	0.35*	0.96***	0.44*	-0.44**	0.41*	0.40**	0.73***	ns	0.54***
Density	1	0.84***	0.84***	0.93***	0.47***	0.93***	-0.89***	0.85***	0.96***	0.76***	0.87***	0.83***
Viscosity		1	0.68***	0.86***	ns	0.84***	-0.97***	0.95***	0.83***	0.47***	0.74***	0.61***
SG			1	0.83***	0.70***	0.85***	-0.63***	0.60***	0.85***	0.83***	0.66***	0.82***
AV				1	0.48***	0.97***	-0.96***	0.96***	0.97***	0.74***	0.74***	0.83***
FP					1	0.56***	-0.51**	0.49**	0.53***	0.82***	0.43**	0.65***
CC						1	-0.96***	0.95***	0.97***	0.80***	0.77***	0.84***
Diesel							1	0.98***	0.89***	-0.73***	0.69***	0.76***
Glycerol								1	0.87***	0.72***	0.65***	0.72***
Ash									1	0.83***	0.82***	0.89***
Na										1	0.72***	0.89***
K											1	0.70***
Mg												1

*Freshness, from clean neat SVOs to NSpVOs , * $p \leq 0.05$, **, $p \leq 0.01$, ***, $p < 0.001$, ns, non-significant*

Useful relationships among the yield and biofuel properties of FAME: Pearson correlation relationships in Table 2 shows freshness of feedstock was strongly related to fuel properties and metal content and also, three were high significant defences in biofuel properties of feedstock and FAME produced. Therefore, as neat VO move to SpVO, there is increasing density, SG, AV, CC and metal content but with decreasing yield of diesel. There was no significant difference in biofuel properties of FAME from neat VOs and SpVOs. Generally, the biofuel properties of generated FAME were strongly correlated with one another, and yield of glycerol, alkali and alkaline metals but were inversely correlated with yield of FAME. Therefore, the higher the viscosity of feedstock /FAME, the higher the other biofuel properties and glycerol content but the lower the yield of biodiesel. The present result was able to statistically reveal the observed differences in biofuel properties, and yield of biodiesel previously reported in similar studies [Barabás and Todorut, 2011; Giakoumis, 2013). High viscosity has been previously linked to high AV, CC, ash content in WVOs due to oxidation, pyrolysis of fatty acids and glycerides to produce oxidative biodegradation and polymerization products that are contaminants in biodiesel (Giakoumis, 2013).

Conclusions: Diversification of spent palm and peanut oils as non-food option for biodiesel production to avoid burden on food security crops and achieve low carbon footprint is well feasible and achievable as experimented in the present study. However, their conversions into neat and clean biodiesel require two steps transesterification other than one step alkali transesterification process. Also, there is need for post two steps transesterification treatment to meet Intentional biodiesel standards. Further investigation was carried out to assess the potential of waste printing paper (WPP) to adsorb metals from the present generated FAME and the results generated shall be reported as Part II in a new paper.

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