A Critical Review of *In-situ* Transesterification Process for Biodiesel Production.

Olusegun D. Samuel, M.Eng.[*1] and Olawale U. Dairo, Ph.D.[2]

[1]Department of Mechanical Engineering, Olabisi Onabanjo University, Ibogun Campus. Via Ifo, Ogun State, Nigeria.
[2]Department of Agricultural Engineering, Federal University of Agriculture, Abeokuta, Nigeria.

E-mail: samolud@yahoo.com[*]

ABSTRACT

A critical review of *in-situ* transesterification process for biodiesel production is reviewed. This article gives an overview of the *in-situ* transesterification, the parameters that have a significant effect on this process, optimization methodologies, as well as advantages and disadvantages of the *in-situ* technique. This will serve as database information for researchers to be in biodiesel production and stake holders.

(Keywords: transesterification, *in-situ*, optimization, biodiesel)

INTRODUCTION

One hundred years ago, Dr. Rudolf Diesel tested peanut oil as fuel for his engine for the first time on August 10, 1893 (Shay, 1993). In the 1930s and 1940s vegetable oils were used as diesel fuels from time to time, usually only in emergencies. However, petroleum fractions that were compatible with the diesel engine became less expensive than vegetable oils, so vegetable oil-based fuels were not commercially viable.

Over the last 20 years, fears for security of supply, increased greenhouse gas emissions (GHG), irresponsible onshore/offshore drilling practices, political instabilities within the oil producing regimes, rising concerns over the continual availability of fusils fuels (Nigeria's cardinal source of foreign exchange), fluctuating oil markets, and the politics of oil trading have resulted in increased interest in vegetable oil-based diesel fuels. Reports have shown that vegetable oils are a possible alternative fuel for diesel engine. However, problems such as injector coking, thickening of lubricants, and oils deposits were recorded on extended operation of diesel engine fuelled with neat or straight vegetable oil (Gupta et al., 2007; Peterson et al., 1990; Pryde, 1983).

Peterson et al. (1987) reported that these problems can be attributed to higher viscosity of the vegetable oil, reduced volatility, and the reactivity of unsaturated hydrocarbon chains. To overcome these constraints, the chemical and physical process like pyrolysis, micro emulsification, and transesterification were especially developed (Ma and Hanna, 1999; Vicente et al., 2007).

A diesel engine could be re-engineered to utilize pure biodiesel oil but this has not yet happened (Srivastara and Prasad, 2000). Until such an engine become widely available, biodiesel refineries convert vegetable oil into an ester, which is similar to diesel fuel, and makes biodiesel usable in the current diesel engines on the market. However, the aforementioned method of reducing viscosity has a drawback. The processes were too expensive for modest through puts, because they were very energy-intensive (Ma and Hanna, 1999). Even though conversion efficiencies are good with conventional transesterification and feed stock costs represent 65 to 75% of the cost of producing biodiesel, there are significant research interests in improving the process and thereby the economics of biodiesel production.

Freedom et al. (1983) reported that ester yields were reduced during conventional transesterification and he attributed it to the existence of gums and extraneous material in the crude vegetable oil. Harrington and D' Arcy – Evans (1985b) researched and claimed that *in-situ* transesterification produced greater yield than the conventional method and both processes yielded similar quantity of fatty acid esters.

Owing to the high market price of edible vegetable oils, conventional transesterification as a means of producing biodiesel is not profitable. To overcome these problems, a myriad of inedible oils are being investigated. The focus of current research is to introduce new transesterification. This technique has to do with direct transesterification of ground oil-bearing seeds with alcohol.

This review critically discusses the *in situ* transesterification process, optimization methodologies, and its advantages as well as disadvantages.

In-situ transesterification is an alternative method of producing ester transport fuels. The process is directed at oil seeds rather than pre-extracted oil, as in conventional transesterification. *In-situ* transesterification is the direct transesterification of ground oil bearing materials instead of purified oils with alcohol and catalyst, to produce alkyl fatty acid esters.

The efficiency or yield of *in-situ* transesterification is defined as the percentage of biodiesel–rich phase over oil content in raw material which is determined by hexane soxhlet extraction. Purity is defined as the percentage of methyl esters in product obtained as the percentage of methyl esters in product obtained from the purification stage. This percentage is mostly obtained from a gas chromatogram result.

MATERIALS AND METHODS

Figure 1 shows conventional versus *in-situ* transesterification. *In-situ* transesterification and conventional processing are depicted below.

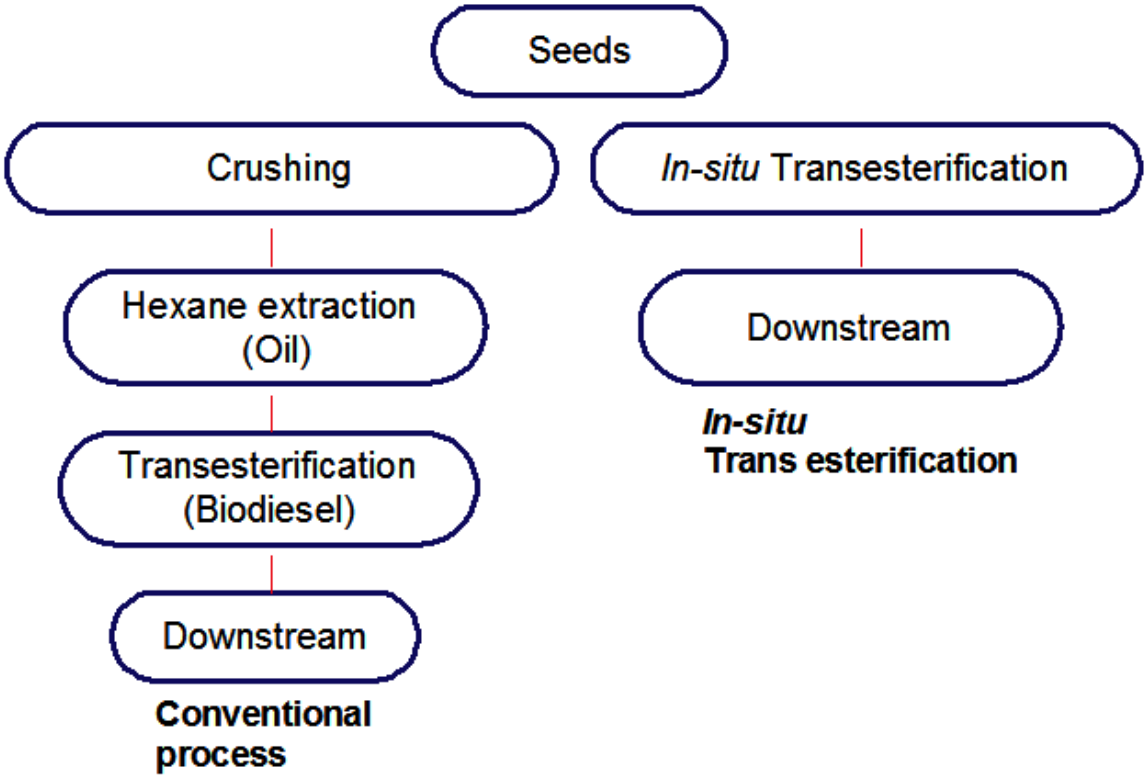

Figure 1: Process Routes for Conventional Production of Biodiesel from Seed (L), and for Direct Extraction-Reaction (R).

It can be seen from Figure 1 that *in situ* transesterification has fewer steps than conventional processing. The crushing and solvent extraction steps that are needed in the conventional process are not employed in the *in-situ* transesterification process. Since the introduction of *in-situ* transesterification by Harrington and D'arcy Evans (1985a), several researchers have investigated the performance and feasibility of this process. Core (2005) emphasized that the possibility of producing biodiesel via *in-situ* transesterification can only be materialized once the process as a whole has been fully characterized.

PARAMETERS IN THE *IN-SITU* TRANSESTERIFICATION

Raw Material

Numerous researchers (Hass et al., 2007; Shuit et al., 2010; and Harvey et al., 2007) reported that an oil bearing seed such as rapeseed, sunflower, Jatropha seeds, and distiller's dried grains with solubles have been studied. The fatty acid profiles of the oils produced by these materials vary substantially. Hence, process parameters differ and these influence biodiesel properties such as cetane number and cold filter plugging point (Ramos et al., 2009). The *in-situ* technique can be applied to almost any oil bearing material (Hass et al., 2007 and Zakaria, 2007).

Catalyst

It was reported (Kildiran et al., 1996 and Qian et. al., 2008) that *in-situ* transesterification is inactive without catalyst. Short–chain alcohols, particularly methanol, are poor solvents for lipids. Zeng et al. (2008) observed that methanol alone was capable of extracting only 4.5% (seed mass) of oil from 20g soya bean, compared with 45% when using n-hexane. Acid or alkali catalyst in *in-situ* transesterification help to breaks the cell walls of oil seeds, thereby facilitating methanol to access the oil in cotyledon cells. Ren et al. (2009) investigated the *in-situ* transesterification of canola via scanning electron microscopy and light microscopy and reported that 95-97% conversion was achieved.

However, in acidic catalyst, the reaction time required when using sulfuric acid to produce 97% yield was 4 hours, while sodium hydroxide only needed 2 hours to produce the same yield. Also, at 40min, 94% of oil had already been converted to methyl ester.

The conversion of oil to methyl esters was typically very high when using methanol and sodium hydroxide. High yields of conversion were achieved by the following researchers. Georgogianni et al. (2008a and 2008b), on sunflower (97%), and Haas et al. (2004) on soybean / methanol / sodium hydroxide (88%). Similarly, Qian et al. (2008) reported of over 95% conversion in reactive extraction of cotton seed during alkaline catalyst. Ability of *in-situ* transesterification was investigated by the Haas group (2004). They reported 91 and 93% conversion on distiller's dried grains with solubles (DDGS) and mean and bone meal (MBM), respectively. Dufreche et al. (2007) observed that 6.23% conversion was achieved using acid catalysis during *in-situ* transesterification of sewage sludge while, 0.38% yield when hexane extraction / acid transesterification was employed. However, when a mixture of hexane, methanol and acetone was utilized to extract the oil, 3.44% yield was obtained. The latter conversion was 2.79% lower and made the variance in validating the economic viability of low or 1 content feed-stocks.

Table 1 highlights different raw materials, catalysts, and solvents used by researchers to produce biodiesel through *in-situ* transesterification (Harrington and D'Arcy – Evans, 1985b; Haas et al., 2007; Shurt et al., 2010; Harvey et al., 2007; Kildiran et al., 1996; Georgogianni et al. 2008; Siler Marinkovic and Tomasevic, 1998, Hass et al., 2004; Georgogianni et al. 2008; Wyatt and Haas, 2009; Oloniyo, 2008; Dairo, 2010). The choice of catalyst very much depends on the feed stock properties, most importantly the concentration of FFA.

Particle Size

Snyder et al. (1984) and Nagy et al. (2008) reported that particle size of the seeds play a very significant factor in conventional solvent extraction Kildiran et al. (1996) invested two sizes of soybeans seed (<1 and <0.5mm) at three different reaction times. He observed that at 1h reaction time, a greater than 1mm particle size

gave the highest percentage of oil dissolved in ethanol, but as the reaction become longer (i.e. at 3 and 5h), smaller size (<0.5) produced better yields.

Ren et al. (2009) reported his finding during the effect of particle size in rape seed *in-situ* transesterification. He observed that as the particle size of the rapeseed fragments increased from 300-500, to 500 – 850, to 1000 – 1400um, the 1h conversion decreased from 86 to 65 to 43% respectively. On the other hand, he highlighted that at the smallest particle size all lipids were removed from the seed particle in 1h. Kaul et al. (2010) also reported of higher conversion during reactive extraction of Jatropha seeds.

Table 1: Different Raw Materials, Catalysts, and Solvents utilized by Researchers to Produce Biodiesel through *In-situ* Transesterification Process.

Raw Material	Solvent	Catalyst	Molar ratio solvent : oil	Reaction time (h)	Temp (°C)	Conversion (Oil basis) (%)	Notes	Ref
Sunflower	Methanol	H_2SO_4 (0.75)	**532:1**	5	65	**93**		Harrignton et al., 1985
Sunflower	Methanol	H_2SO_4 (0.7)	**300:1**	4	64.5	**98.2**		Silver-Marinkov and Tomasevic, 1998
Soybean	Methanol	H_2SO_4 (0.75)	**281:1**	10	65	**23.3**		Kildiran et al., 1996
Soybean	Methanol	H_2SO_4 (0.75)	**150:1**	3	121	**83**	CO_2 cosolvent	Wyatt and Haas, 2009
Jatropha curcas	Methanol	H_2SO_4 (0.2)	**300:1**	24	60	**99.8**	Hexane cosolvent	Shuit et al., 2010
Microbial Biomass	Methanol	H_2SO_4 (0.2)	**830:1**	20	70	**96.8** (Lipomyces starkey) **91.0** (Mortierella isabellina) **98.1** (Rhodosporidum toroloides)		Liu and Zhao, 2007
Primary sewage sludge	Methanol	H_2SO_4 (0.9)	**1400:1**	24	75	**66**		Mondala et al., 2009
Soybean	Methanol	NaOH (0.09)	**543:1**	8	23	**88**		Haas et al., 2004
DDGS	Methanol	NaOH (0.4)	**655:1**	1.2	35	**91.1**		Haas et al., 2007
MBM	Methanol	NaOH (2.0)	**550:1**	0.2	35	**9.3**		Haas et al., 2007
Cottonseed	Methanol	NaOH (0.4)	**673:1**	0.3	60	**95**	Ultrasound	Georgogianni et al., 2008a
Cottonseed	Ethanol	NaOH (0.4)	**613:1**	0.7	80	**98**	Ultrasound	Georgogianni et al., 2008a
Sunflower	Methanol	NaOH (0.4)	**476:1**	0.7	60	**97**	Ultrasonic	Georgogianni et al., 2008b
Sunflower	Ethanol	NaOH (0.4)	**434:1**	0.7	80	**98**	Ultrasonic	Georgogianni et al., 2008b
Sunflower	Methanol	NaOH (0.2)	**101:1**	13	20	**98**	DEM cosolvent	Zeng et al., 2008
Jatropha curcas	Methanol/ ethanol mix	NaOH (0.02)	**512:1**	1	60	**87**		Hervey et al. 2007
Jatropha curcas	Methanol	NaOH (0.04)	**100:1**	1	60	**70**		Oloniyo, 2000
Castorbean	Ethanol	NaOH (0.3)	**100:1**	1.4	60.3	**99.5**		Dairo, 2010
Jatropha curcas	Ethanol	NaOH (0.75)	**100:1**	1.5	60	**78**		Amusan, 2010
DDG: Distillers dried Grains with solubles; Dem: Diethoxymethane; MBM: meat and Bone meal, Temp: Temperature								

The observations of researchers indicated that smaller seed particles size increases rate of reaction, indicating that the rate is controlled by internal mass transfer (Ren et al., 2009).

Moisture Content

Ma and Hanna (1999) reported that presence of water in conventional transesterification reduced the potential yield of the methyl and difficulty in separation between the glycenl and alkyl ester – rich phase. Hass et al. (2007b) observed that the quantity if alcohol required during *in-situ* transesterification was significantly lessened in dried seeds. They reported 60% reduction of methanol and 56% reduction of sodium hydroxide when soybean flakes were dried in a convention oven until it had a water content of 0%. Experiments at 2.6% water content samples reduced the methanol and sodium hydroxide requirement by 40 and 33%, respectively.

Qian et al. (2008) reported of significant increment in methyl ester conversion from 80 to 98% when the moisture content was reduced from 8.7 to 1.9%. Zakaria (2007), however, reported that *in-situ* transesterification of grounded rape seed using methanol/sodium hydroxide with drying seeds from 6.7 to 0 wt% water does not reduce the solvent requirement, nor increase the yield of ester. It was realized that the ester yield only reduced when there was more than 2wt% water in the solvent.

Molar Ratio of Alcohol to Oil

Numerous researchers agree that in the process of either acid-catalyzed or alkali-catalyzed *in situ* transesterification, alcohol/oil molar mass was which higher than the value calculated according to stoichiometry of the transesterification reaction (Zeng et al., 2009). For instance, Siler-Marinkovic and Tumasevic (1998) used a 300:1 ratio in their experiment with sulfuric acid catalyst, while Haas et al. (2004), Evans (1985b) and Siler-Marinkovic and Tomasevic (1998) reported yields of 93.2 and 98.2% respectively when they used methanol to oil ratios of 370 and 300:1.

Co-solvents have been reported by researchers (Zeng et al. 2009; Mao et al., 2004 and Boocock et al., 1998) to improve the solubility of alcohol and accelerate the *in-situ* transesterification.

Alcohol Type

Various mono hydroxide alcohols can be employed in the *in-situ* transesterification method. The usage of higher alcohols reduced the biodiesel's purity and these alcohols tend to be more expensive.

Types of Catalyst in *In-situ* Transesterification

Acid and Alkali Catalysts: The factors which influenced the acid catalyzed *in-situ* transesterification, such as molar ratio of alcohol/oil, catalyst amount, reaction time, and temperature, have been investigated by different researchers and they reported that the overall reaction rate of the *in-situ* transesterification was mainly determined by the parameters affecting the extraction rate (Ozgul and Turkay, 2002, 2003; Haas et al., 2004).

Considering that the acid-catalyzed *in-situ* transesterification was a time-consuming process, Haas et al. (2004) developed an alkali catalyzed *in-situ* transesterification process. Amount 95% of the total oils were extracted from the raw material, and 84% of those oils were converted into biodiesel dropped under optimal conditions. When the moisture content of flaked soybean dropped from 7.4% to 2.6%, the volume of methanol required to convert 5g of soybean flakes decreased from 30ml to 18ml, respectively (Haas et al., 2007). They further reported that drying the soybean flake resulted in a marked reduction in reagent requirement for transesterification, stressing the effect of moisture removal on *in-situ* transesterification.

Silver-Marin Kovic and Tomosevic (1988) reported that the biodiesel from sunflower seed and the properties of esters produced was observed to have met the ASTM standards for biodiesel.

Optimization of Transesterification process for Biodiesel Production

Optimization of experiment has been reported by Robert et al. (2002) as a way of experimentation leading to useful saving of scientific resources. Several scientific decision of experiments method can be utilized to explore which variables and at

what level of the variables will maximize a particular output.

Numerous researchers have embarked in optimize power variable in the production of biodiesel from various crops both conventional and *in-situ* (Alamu et al., 2007; Dairo, 2010, Satharornvichit et al., 2006). Optimization procedures usually applied are factorial designs, response surface methodology, uniform experimental design, tagorch, orthogobal and several others.

Biodiesel Quality

One of the most significant factors to be considered in the development of *in-situ* transesterification is whether the process can provide the market with quality biodiesel and meet the requirement of governing bodies. Two of the most accepted standards are ASTM D 6751 and EN 14214.

Advantages and Disadvantages of *In-situ* Transesterification

- The simplicity of the process will pave for oil seed growers from overdependence on crushing and solvent extraction plants.
- Certain features of the process will afford the producers in rural areas to produce their own fuels.
- The process presents with huge challenges in order to make profits, especially on how to reduce the volume of an alcohol in the reaction.

CONCLUSION

A review of the literature has now show that numerous studies were conducted on the process parameters on the *in-situ* transesterification of several oil bearing seeds such as sunflower, soybean, jatrophe, cotton seed and sludge. Different catalysts and solvents were also used on the *in-situ* technique by different researchers.

Conversion of biodiesel quality and purity of the biodiesel were also assessed by gas chromatography. The research results indicate that *in-situ* transesterification can be used effectively to produce biodiesel, the optimum process factors levels could also be used in scale-up biodiesel equipment plant. This is possible if researchers can reduce the volume of alcohol in the reaction. In addition, technical feasibility has been demonstrated for a range of feedstock, catalysts and alcohols.

REFERENCES

1. Alamu, O.J., M.A. Waheed, and S.O. Jekayinfa. 2007. “Alkali – Catalyzed Laboratory Production and Testing of Biodiesel Fuel from Nigerian Palm Kernel Oil”. *Agricultural Engineering International: The CIGR E Journal.* Manuscript Number EE 07009. 9.
2. Amusan, O.O. 2001. “*In-situ* Transesterification of Jatropha Seeds for Biodiesel Production”. Unpublished B. Eng. Thesis. Dept. of Agricultural Engineering, UNAAB: Nigeria. 1-50.
3. Antolin, G., F.V. Tinaut, Y. Briceno, V. Castono, C. Peres, and A. Ramirez. 2002. “Optimisation of Biodiesel Production by Sunflower Oil Transesterification”. *Bio-resource Technol*. 83: 111-114.
4. Boocock, D.G.B., S.K. Komar, V. Mao, and H. Sidi. 1998. “Fast One–Phase Oil-Rich Process for the Preparation of Vegetable Oil Methyl Esters”. *Biomass Bioenergy*. 11:43-50.
5. Core, J. 2005. “New Method Simplifies Biodiesel Production”. Agricultural Research Services: Washington, D.C. 13.
6. Dairo, O.U. 2010. “*In-Situ* Transesterification of Caster Oil Bean Seed into Biodiesel using a Batch Processor”. Unpublished Ph.D Thesis. Dept. of Agricultural Engineering, UNAAB: Nigiera. 1 -204.
7. Dufreche, S., R. Hernandez, T. French, D. Sparks, M. Zappi, and E. Alley. 2007. “Extraction of Lipids from Municipal Waste Water Plant Micro Organisms for Production of Biodiesel”. *Journal of Am. Oil. Chem Soc.* 84(2): 181–187.
8. Freedman, B., E.H. Pryde, and T.L. Mounts. 1983. “Variables Affecting the Yields of Fatty Esters from Transesterified Vegetable Oils”. *J. Am. Oil Chem. Soc*. 61(10):1638 – 1643.
9. Georgogianni, K.G., M.G. Kontominas, P.J. Pomons, D. Avlonitis, and V. Gergis. 2008a. “Alkaline Conventional and *in situ* Transesterification of Cotton Seed Oil for the Production of Biodiesel”. *Energy Fuel.* 22(3):2120-2115.

10. Georgogianni, K.G., M.G. Kontominas, P.J. Pomonis, D. Avlonitis, and V. Gergis. 2008b. "Conventional and *in situ* Transesterification of Sunflower Seed Oil for the Production of Biodiesel". *Fuel process Technol*. 89(5):503 – 509.

11. Gupta, P.K., R. Kumar, B.S. Panesar, and V.K. Thapar. 2007. "Parametric Studies on Biodiesel Prepared from Rice Bran Oil". *Agricultural Engineering International: The CIGR Journal of Scientific Research and Development*. IX (EE06007).

12. Haas, M., K. Scott, T. Foglia, and W. Mariner. 2007b. "The General Applicability of *in situ* Transesterification for the Production of Fatty Acid Esters from a Variety of Feedstocks". *J. Am. Oil Chem. Soc.* 84 (10); 96 3-970.

13. Haas, M.J., K.M. Stock, W.N. Marner, and T.A. Foglia. 2004a. "*In situ* Alkaline Transesterification: An Effective Method for the Production of Fatty Acid Esters from Vegetable Oils". *J. Am. Oil Chem. Soc.* 81(1):83-89.

14. Hass, M.J. and K.M. Scott. 2007c. "Moisture Removal of *in situ* Biodiesel Production from Soybeans". *J. Am. Oil Chem. Soc.* 81(2):197 – 204.

15. Harrignton, K.J. and C. D'Arcy – Evans. 1985a. "Transesterification *in situ* of Sunflower Seed Oil". *Ind. Eng. Chem. Prod Res Dev*. 24(2):314 – 318.

16. Harrignton, K.J. and C. D'Arcy–Evans. 1985b. "Comparison of Conventional and *in situ* Methods of Transesterification of Seed Oil from a Series of Sunflower Cultivars". *J. Am. Oil Chem. Soc.* 02 (6), 1009 – 1013.

17. Harvey, A.P., R. Khurana, and J.G.M. Lee. 2007. "*In-situ* Transesterification of Jatropha Seeds to Produce Biodiesel using Alcohol Mixture". Presented at: 4th international Bio fuels Conference: New Delhi, India.

18. Khalil, N.C. and L.C.F. Leite. 2006. "Process for Producing Fuel using Triglycerides – Rich Oleaginous Seed Directly in a Transesterification Reaction in the Presence of an Alkaline Alkoxide Catalyst". US Patent No. US 7, 112, 22a B2.

19. Kildiran, G., S. Yucel, and S. Tunkay. 1996. "*In situ* Alcoholysis of Soybean Oil". *J. Am. Oil Chem Soc.* 73(2):225 – 228.

20. Liu, B. and Z. Zhao. 2007. "Biodiesel Production by Direct Methanolysis of Oleaginous Microbial Biomass". *J. Chem. Technol. Bio Rechenol*. 82(8):775 – 780.

21. Ma, F. and M.A. Hanna. 1999. "Biodiesel Production: Review". *Bioresource Technol*. 70(1):1-15.

22. Mao, V., S.K. Konar, and D.G.B. Boocock. 2004. "The Pseudo-Single Phase, Basic Catalysed Transemethylation of Soybean Oil". *Journal of American oil Chemists Society".* 81:803-808.

23. Mondala, A., K. Liang, H. Toghiani, R. Hernandez, and T. French. 2009. "Biodiesel Production by *in situ* Transesterification of Municipal Primary and Secondary Sludges". *Bioresource Technology*. 100 (3):1203 – 1210.

24. Nagy, B. and B. Siminandi. 2008. "Effects of Particle Size Distribution Moisture Content, and Initial Oil Content on the Super Critical Fluid Ectraction of Paprika". *Journal of Super Critical Fluids*. 46(3):293 – 298.

25. Oloniyo, O. 2008. "Reactive Extraction of Biodiesel from Jatropha". MSC CREM, New Castle University, UK.

26. Ozgul Yucel, S. and S. Turkays. 2002. "Variables Affecting the Yields of Methyl Esters Derived from *in situ* Esterification of Rice Bran Oil". *Journal of American Oil Chemistry Society.* 79(6):611 – 614.

27. Ozgul Yucel, S. and S. Turkays. 2003. "Fa Mono Alkylesters from Rice Bran Oil *in situ* Esterification". *Journal of American Oil Chemistry Society*. 80(1):81 – 84.

28. Peterson, C.L., R.O. Cnz, L. Perkings, R. Konis, and D.L. Auld. 1990. "Transesterification of Vegetable Oil for use as a Diesel Fuel: A progress Report". ASAE paper No. 90-610.

29. Peterson, C.L., R.A. Koris, P.G. Mora, and J.P. Madson. 1987. "Fumigation with PNpane and Transesterification Effect on Injector Cooking with Vegetable Oil". *Transactions Am. Soc. Agricultural Engineers*. 30(1):28 – 35.

30. Pryde, E.H. 1983. "Vegetable Oil as a Diesel Fuels: Overview". *Journal of American Oil Chemistry Society*. 60(8):1557 – 1558.

31. Qian, J., F. Warg, S. Liu, and Z. Yun. 2008. "*In situ* Alkaline Transesterification Seed Meal". *Bio Resource Technol.* 99(18):9009 – 9012.

32. Ramos, M.J., C.M. Fernandez, A. Casas, Z.L. Rodriguez, and A. Peres. 2009. "Influence of Fatty Acid Composition of Raw Materials on Biodiesel Properties". *Bio Resource Technol*. 100(1):261 – 268.

33. Ren, Y., R. Zakaria, and A.P. Harvey. 2009. "Towards a Biodiesel – Based Bio Refinery

Chemical and Physical Characterization of Reactively Extracted Canola". Presented at: International Bio refineries conference (IBC). New York, NY.

34. Robert, S., F.W. Micheal, I.P. Festing, and F. Larry. 2002. "Use of Factoral Design to Optimize Animal and Experiments to Reduce Animals". *ILAR, Journal – Experimental Design Statistic in Biomedical Research.* 43(3):15.

35. Shay, E.G. 1993. "Diesel Fuel from Vegetable Oils Status and Opportunities". *Biomass and Bioenergy* 4(4):227 – 242.

36. Shuit, S.H., K.T. Lee, A.H. Kamaruddin, and S. Yusup. 2010. "Reactive Extraction and *in situ* Esterification of *Jatropha curcas* Seeds for the Production of Biodiesel". *Fuel.* 89(2):527 – 530.

37. Siler–Marinkovic, S. and A. Tumaseric. 1998. "Transesterification of Sunflower Oil *in situ". Fuel*. 77(12):1389 – 1389.

38. Sathavorvichit, N., P. Bookkamana, and B. Publin. 2006. "Central Composite Design in Optimization of the Factors of Automatic Flux Cored Arc Welding for Steel ST37". *Proceedings of the 2nd IMT – GT Regional Conference on Mathematics, Statistics and Applications.* Unicersity Sains Malaysia , Penang, June 13 – 15.

39. Snyder, J.M., J.P. Friedrich, and D.D. Christiansoon. 1984. "Effect of Moisture and Particle Size on the Extractability of Oils from Seeds with Super Critical Co_2". *Journal of American Oil Chemistry Society*. 61(12):1851 – 1856.

40. Srivastava, A. and R. Prasad. 2000. "Triglycerides–Based Diesel Fuels". *Renewable and Sustainable Energy Review.* 4:111 – 133.

41. Vincete, G., Z. Martine, and J. Aracil. 2007. "Optimization of Integrated Biodiesel Production. Part II: A Study of Material Balance". *Bio resource Technology.* 98:1754 – 1761.

42. Wyatt, V.T. and M.J. Haas. 2009. "Production of Fatty Acid and Methyl Esters via the *in situ* Transesterification of Soybean Oil in Carbondioxide – Expanded Methanol. *Journal of American Oil Chemistry Society*. 86(10):1009 – 1016.

43. Zakaria, R. 2007. "Reactive Extraction of Bbiodiesel". First year Ph.D report. CEAM, New Castle University: UK.

44. Zeng, J. and Jiangil. 2008. "Acis Catalyzed Esterification of *Zanthoxylum bungeanum* Seed Oil with High Free Fatty Acids for Biodiesel Production". *Bio Resource Technol.* 99:8995 – 8998.

45. Zeng, J.L., X.D. Wang, B. Zhao, J. Sun, and Y.C. Wang. 2009. "Rapid *in –situ* Transesterification of Sunflower". *Ind. Eng. Chem. Re.* 48: 850 – 856.

ABOUT THE AUTHORS

Engr. Olusegun David Samuel, is a Lecturer at the Department of Mechanical Engineering. He is a member of the Nigerian Society of Engineer, (NSE), International Association of Engineers (IAENG), Association of Maintenance and Design Engineers (ASSOMEDEG), and a Registered Engineer (COREN). He is currently undertaking his Ph.D. degree at the University of Agriculture, Abeokuta, Ogun State, Nigeria. He holds an M.Eng. degree in Mechanical Engineering. His research interests are in renewable energy, manufacturing, production, residual stress, lubrication technology, and engineering.

Dr. O.U. Dairo, is a Senior Lecturer in the Department of Agriculture Engineering, Federal University of Agriculture, Abeokuta, Nigeria. He is a Registered Engineer (COREN) and is a member of the Nigerian Society of Engineer, (NSE). He holds a Ph.D. degree in Agricultural Engineering. His research interests are in the utilization of crops as alternatives farm energy sources using under-exploited seeds and design of biodiesel processors.

SUGGESTED CITATION

Samuel, O.D. and O.U. Dairo. 2012. "A Critical Review of *In-situ* Transesterification Process for Biodiesel Production". *Pacific Journal of Science and Technology*. 13(2):72-79.

Pacific Journal of Science and Technology