Ultrasonic Comparative Assessment for Biodiesel Production from Rapeseed

N.N. Ibiari*, S.A. Abo El-Enin, N.K. Attia and G. El-Diwani

Chem. Eng. and Pilot Plant Dept., National Research Centre, Egypt

Abstract: The application of ultrasound during extraction and trans-esterification of oil from rapeseed was evaluated. Two methods of extraction were used, batch wise extraction and soxhlet extraction. In batch wise extraction procedure, ground rapeseeds were added to solvent and ultra-sonicated either by cleaning bath or ultrasonic generator. Conventional soxhlet extraction assisted in the soxhlet chamber by ultrasound has been developed. Ultrasonic technique reduced time required to extract oil. Using batch wise extraction procedure, percent recovery of oil increased almost 17.83% and 20.99% by using cleaning bath and ultrasonic generator respectively rather than control after 2hrs.While in using soxhlet extraction percent recovery reached 85% after 1.5 hr in case of ultrasonic and after 4 hrs without using ultrasonic. Physical and chemical properties of rapeseed oil were tested. Then the alkaline trans-esterification of rapeseed oil with methanol and potassium hydroxide for production of biodiesel was studied, using ultra-sonication and magnetic stirring. In trans-esterification the use of ultra-sonication and magnetic stirring led to similar high yields of 90% of methyl esters after approximately 10 min. of reaction time. Comparison between biodiesel obtained and standard biodiesel and diesel fuel was done.

[N.N. Ibiari, S.A. Abo El-Enin, N.K. Attia and G. El-Diwani. **Ultrasonic Comparative Assessment for Biodiesel Production from Rapeseed**. Journal of American Science 2010;6(12):937-943]. (ISSN: 1545-1003). http://www.americanscience.org.

Keywords: Ultrasound, extraction, biodiesel, rapeseed oil, transesterification.

1. Introduction:

Diminishing fossil fuel resources coupled with the steady increase in energy consumption have spurred research interest in alternative and renewable energy resources. Bio-diesel is a promising nontoxic and biodegradable renewable fuel comprised of mono-alkyl esters of long chain fatty acids, which are derived from vegetable oils (edible and non-edible)(Hanna 1999) . Seeds of high oil content such as sunflower, soybean and canola as edible oils, and jatropha and rapeseed as non-edible oils used as feed stock for bio-diesel production.

Extraction is one of the key processing steps in recovering oils contained in seeds. Mechanical pressing (Abu- Arabi 2000) is the simplest method of extraction, however, needs no extraction medium. It has been traditionally applied to the extraction of oils from oil seeds; the only equipment needed is a hydraulic press.

Traditional extraction processes may be classified as follows: extraction with organic solvents: percolation, maceration, and extraction using a Soxhlet apparatus; and extraction with water: infusion, decoction, and steam distillation (Silva 1998). An old method also worth mentioning is extraction with cold fat, called effleurage, used mainly for extraction of fragrances from flowers (Starmans 1996) These techniques are often time consuming and require large volume of organic solvent.

In last decade, alternative extraction techniques (non conventional extraction methods) that introduce some form of additional energy to process in order to facilitate the transfer of analysts from sample to solvent have been considered. These methods include ultrasound assisted extraction, pressurized liquid extraction, microwave extraction and supercritical fluid extraction {Huie 2002, Flores 2006, Garcia 2004, Vinatoru 2001} ,as well as vertical (turbo) extraction. The main advantage of these non-conventional methods compared to conventional methods is the increased extraction efficiency, which leads to increased yield and/or shorter extraction time.

The ideal approach would be one retaining the advantages of soxhlet extraction (namely, sample fresh solvent contact during the whole extraction step, no filtration step, simple manipulation) while circumventing its short comings by accelerating the process and minimizing environmental pollution.
With this aim, an ultrasound-assisted soxhlet extractor has been designed and constructed. The device is based on the same principles as the conventional soxhlet extractor but modified in order to allow location of the soxhlet chamber in a bath through which ultrasounds are applied by means of an ultrasonic probe. The same approach was done with the wise batch extraction.

The interest in the use of renewable fuel started with the direct use of vegetable oils as a substitute for diesel (Demirbas 2003). However, their direct use in compression ignition engines was restricted due to high viscosity, low volatility and polyunsaturated character (Ramadhas 2005). To overcome these constrains there are at least four ways in which oils and fats can be converted into bio-diesel (Ghadge 2006), transesterification, blending, micro emulsion (Fukuda 2001)and pyrolysis. Out of these methods, trans-esterification is the most viable process. The trans-esterification process is achieved by reaction of triglyceride molecule with an excess of alcohol in the presence of a catalyst to produce glycerin and a fatty acid methyl esters "FAME" which is the bio-diesel.

Stochiometrically, three moles of alcohol are required for each mole of triglyceride, but in general, a higher molar ratio is often employed for maximum ester production depending upon the type of feed stock, amount of catalyst, temperature etc. However, the yield of bio-diesel is independent of the type of the alcohol used (methanol, ethanol, propanol and butanol) (Sharma 2008) and the selection of one of these depends on cost and performance. Methanol is preferred over others due to its low cost (Ramadhas 2005) and its physical and chemical advantages such as polar and shorter-chain alcohol (Hanna 1999). The conventional catalysts used are acid and alkali catalysts depending on the nature of the oil used (free fatty acids FFA content in the raw oil) (Fukuda 2001, Freedman 1986). FFA should not exceed a certain amount for trans-esterification to occur by an alkali catalyst (Canakci 1999, 2001).In case of high free fatty acid, acid esterification using H_2SO_4 as a catalyst was done to reduce the FFA prior to alkaline transesterification [Ramadhas 2005, Veljkovic 2006, Sahoo 2007, Shanna 2007) . The common catalyst employed during alkaline transesterification includes homogeneous catalyst such as sodium hydroxide, potassium hydroxide and sodium methoxide (CH_3ONa) (Leung 2006). Sodium methoxide proved to be more effective than NaOH, which produces a small amount of water by mixing NaOH and methanol. However, NaOH and KOH are widely used in the industrial biodiesel production process owing to its low cost (Jeong 2004). The amount of alkali catalyst was calculated on the basis of the amount needed to neutralize the unreacted acids plus 0.35% for virgin oil which came out to be 0.55% w/v KOH (Tiwari 2007).

Presence of sufficient amount of methanol during the transesterification reaction is essential to break the glycerin-fatty acid linkages (Widyan 2002). But excess of methanol should be avoided. Increasing the molar ratio of methanol/oil beyond 6:1 neither increases the product yield nor the ester content, but rather makes the ester recovery process complicated and raised its cost.

Lifka and Ondruscka (Lifka 2004) said that since basic transesterification showed no significant temperature dependence, energy required for thermo starting the reaction mixture can be saved. From the energy balance of the three mixing methods (magnetic stirrer, ultrasound and ultra turrax) employed, it was determined that energy costs are lower for ultrasonic mixing.

The main objective of the present work is to compare classical extraction techniques of oil (Soxhlet, batch wise extraction) with ultrasound-assisted extraction method, and to apply the biodiesel production technology using an alkaline catalyst to the transesterification of rapeseed oil and particularly to study the effect of ultrasonication versus mechanical stirring on methyl esters yield and reaction time.

2. Materials and methods

2.1 Materials:

- Rape Seeds used in this work were supplied by Cultivation and Production of Medicinal and Aromatic Plant Dept. at National Research Centre of Egypt.
- Solvent used for oil extraction was commercial hexane, obtained from Alexandria Petroleum Company of Egypt.
- Methanol of 99.8% analytical reagent.
- Potassium hydroxide pellets of 98% purity as catalyst.

2.2 Rape Seed Preparation:

The rape seeds were milled in an electric mill, sieved and classified into three different particle size groups ≤234, ≤600 and ≤850 µm. Moisture content of rape seeds was determined.

2. 3 Analytical Methods:

Moisture and oil content in seeds were determined gravimetrically. The fatty acid composition of rapeseed oil was determined using gas chromatographic analysis of the oil ethyl esters. Modification of the oil to its ethyl esters was made using 2% H_2SO_4 as catalyst in the presence of dry ethyl alcohol in excess. The chromatographic analysis was made using Hewlett Packard Model 6890 Chromatograph. A capillary column 30 m length and 530 µm inner diameters packed with Apiezon® was used. Detector temperature was 280°C, injection temperature was 300°C and the column temperature was programmed from 100 to 240°C at 15°C/min.

Viscosity was measured using Brookfield Viscometer Model DV-II+. The acid values are milligrams of KOH necessary to neutralize the free acidity in 1g of oil sample.

Acid Value = {(Titration (ml) *5.64) /wt of sample used} (mg KOH/g)

2.4 Extraction Procedure

Extraction was done by two different methods as shown in Fig.1 .

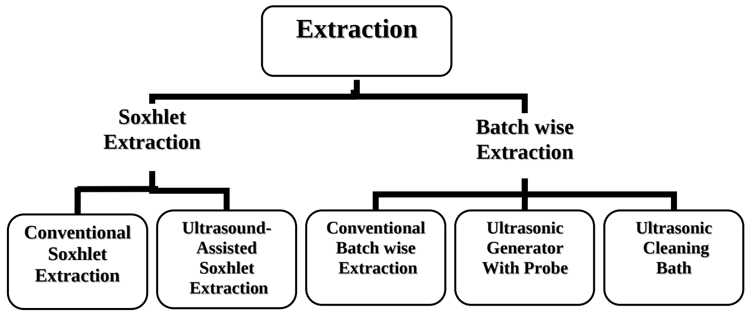


Fig (1): Extraction Methods

2.4.1 Soxhlet Extraction:

As shown in Fig. 2, the apparatus of conventional soxhlet extraction was modified by adding a water bath with an orifice at the bottom in order to enable connection with a distillation flask. A titanium alloy ultrasonic probe (2.75 mm diameter) was immersed in the water bath and used to accelerate the extraction process.

2.4.2 Batch –Wise extraction process:

2.4.2.1Conventional Extraction

Ground rape seeds are mixed in an glass reactor with hexane. Suspensions were continuously stirred at constant temperature and stirring rate (using a magnetic stirrer) for different time intervals. Suspension is filtered and extracted oil was separated from the solvent using rotary vacuum evaporator.

2.4.2.2 Ultrasonic Generator with Probe Assisted Extraction

Same steps as in conventional method except that the suspension was ultra-sonicated during extraction time using ultrasonic generator probe which was submerged in the suspension. Temperature was kept constant during sonication and extraction.

2.4.2.3 Ultrasonic Cleaning Bath Assisted Extraction

Ground rape seeds and hexane as extracting solvent were mixed in a closed flask. The sonication was performed at constant temperature for different time intervals using an ultrasonic cleaning bath.

A rotary – evaporator was used to release the solvent after each extraction.

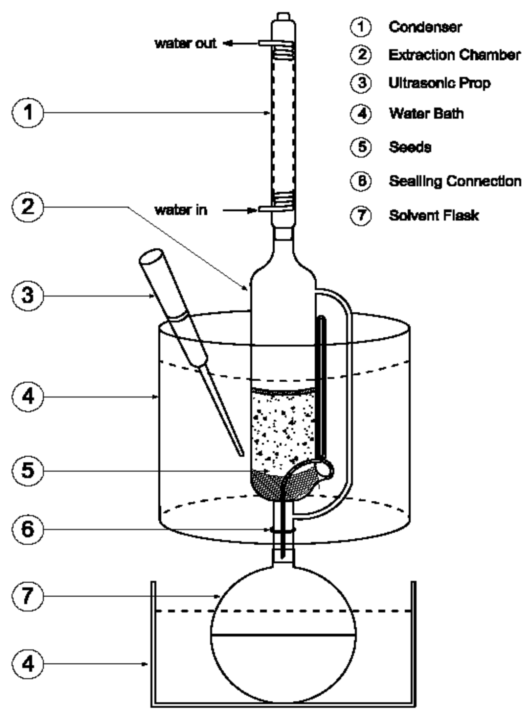


Fig (2): Soxhlet extraction apparatus

2.5 Transesterification Procedure:

Rapeseed oil produced, methanol corresponding to 6:1 molar ratio of alcohol to oil and KOH (1% w/w) were refluxed together in a glass reactor placed in water bath. Heating was achieved by means of hot plate with temperature controller. The temperature was raised to 65°C and the mixture was stirred either using a magnetic stirrer or an ultrasonicator (ultrasonic generator with probe or ultrasonic cleaning bath).

After different reaction time intervals, the reaction was stopped and the mixture was allowed to stand for phase separation in a separating funnel. The esters mixture formed the upper layer and glycerin formed the lower layer (Stavarache 2005)

3. Results ands Discussion:

3.1. Effect of moisture content on the percentage yield of extracted oil

No significant effect in the percent oil extracted from dried seed (38.16%) and non dried seed (38.68%) using soxhlet extraction. This may be attributed to the moisture content range from 5.86 to 6%, which is less than the moisture level between 8 and 20% for optimum yield when harvesting (Loren 2007)

3.2. Effect of particle size on the percentage yield of extracted oil

The effect of seeds' particle size on the oil yield was studied by soxhlet extraction of three classes of ground rape seeds using hexane.

As can be seen from Table (1) the highest oil yield was obtained from the higher class of the ground rape seeds. Thus in the further research, the higher class of ground rape seeds were used.

Table (1) Effect of Particle Size on Extracted Oil Yield

Class	*Particle Size, mm*	*%Yield of oil*
Largest	**0.6-0.85**	**36.52**
Medium	**0.6**	**31.3**
Smallest	**0.234**	**26**

3.3. Effect of Time on the percentage yield oil extracted:

3.3.1. Using Soxhlet Extraction

The percent oil recovery by conventional soxhlet extraction and ultra-sonic assisted soxhlet extraction (UASE) with hexane using different extraction time are compared in Fig. (3). It is clear that using UASE reduces the time of extraction from about 4hr to 1.5 hr, but the percent recovery is slightly lower and this is due to the high frequency (800 KHz) ultrasound employed. When the high frequency ultrasound is employed the extraction yield did not increase significantly, however the degradation of the seed constituents was diminished. In case of low frequency sonication degradation becomes more important (Vinatoru 2001).

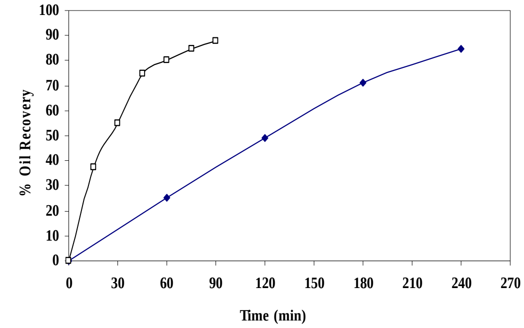
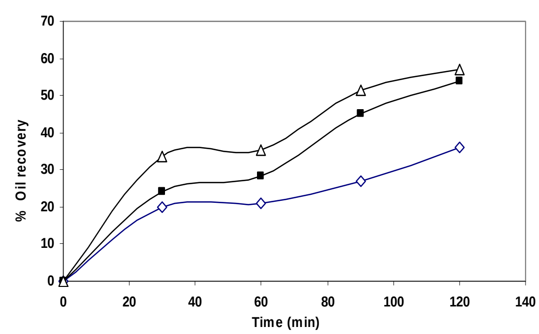


Fig (3):Effect of time on percentage recovery using soxhlet and UASE

soxhlet soxhlet+sono

3.3.2. Using Batch Wise Extraction Process

The oil extraction capabilities of three different methods (control, ultrasound cleaning bath and ultrasound probe) using hexane (seed: solvent (1:10 ratio)) at different extraction time are shown in Fig (4). The percentage of oil recovery is increased with application of ultra-sound. But the relative increase in the percent of oil recovery of the two methods of ultrasound differed. For sonic bath, the percent recovery increased by 4.35% at an extraction time of 30 min and 17.83% at an extraction time of 2 hr. While in case of using probe, the percent recovery increased by 13.47% at an extraction time of 30 min and 20.99 at an extraction time of 2 hr.



Fig(4):Effect of time on percent recovery using batch wise extraction process

control sonic bath sonic probe

It is interesting that the higher yield of extracted oil achieved using soxhlet extraction technique either with or without ultrasonic than using batch wise extraction process. This can be attributed not only to a higher operating temperature and much longer extraction time, but also to a higher seed – solvent ratio applied in the Soxhlet extraction.

3.4 Effect of type of extraction on the properties of rapeseed oil

In order to evaluate if the oil composition was affected during ultrasound assisted extraction, gas liquid chromatography analyses were carried out on two different extracted samples using UASE and soxhlet. Table (2) shows that no appreciable differences in the extracts composition. This indicates that the oil composition is not affected by the use of ultrasound, and these results agree with Luque-Garcia J.L. et al (Luque 2004).

Table (2) Fatty acid composition of rapeseed oil

Constituent	% composition using UASE	% composition using soxhlet extraction
Palmitic (16:0)	4.34	4.39
Stearic (18:0)	1.55	1.67
Oleic (18:1)	60.0	60.95
Linoleic (18:2)	20.0	19.04
Linolinec (18:3)	12.5	11.78
Eicosic (20:1)	27.0	27.1
Erucic (22:1)	0.9	0.9

Average molecular weight =882.6 k.mol/kg

Characterizations of rapeseed oil are shown in Table (3).

Table (3) Characterization of rapeseed oil

Viscosity, (c.ps) at 40 °C	46.5
Free fatty acid, %	1.5
Acid value, (mg NaOH/1gm oil)	0.3

3.5 Transesterification:

A set of experiments was carried out to determine the effect of magnetic stirring and ultrasonication on the transesterification reaction. After 10 min, conversions of more than 90% were obtained. So no significant influence due to the mixing method was observed. Percent conversion to ester values is higher than those of Lifka and Ondruscka [26] who reported more than 85% conversion of rapeseed oil to methyl esters after 10 min. at 45̊C, on the alkaline transesterification of rapeseed oil using NaOH at a concentration of 1 % w/w, using different mixing methods (magnetic).

Table (4): Comparison of different standard of biodiesel in worldwide with produced biodiesel from rapeseed and conventional diesel.

Property	Units	Biodiesel (Rapeseed)	US National Biodiesel	German Biodiesel	Conventional Diesel
Flash point	̊ C	163	100	110	54
Pour point	̊ C	-6			
Cloud point	̊ C	-3			
Kinematics viscosity at 40˚ C	mm^2/s	6.08	1.9-6	3.5-5	2.3
Density at 15˚ C	Kg/m^3	884	860	875-900	800
Carbon Residue	% (w/w)	0.258		0.3	0.3

Table (4) represents the characterization of biodiesel obtained from rapeseed and standards of biodiesel and diesel fuel in worldwide.

Flash point is the lowest temperature at which the vapor realized by a liquid, can form an ignitable mixture with air. Flash point of a fuel is a security parameter, with great importance for its transport and storage. The flash point of biodiesel is one of its main advantages (163 ̊ C) in our case when compared to diesel fuel (54̊ C).

Cloud point data consistently over predict the cold temperature limit at which start-up or performance problems may be expected to occur. The temperature at which crystal agglomeration is extensive enough to prevent free pouring of fluid is determined by measurement of its pour point

(Hochauser 1994).Feed stocks with relatively low concentrations of saturated long-chain fatty acids generally yield biodiesel with much lower cloud point and pour point. Thus, feed stocks such as linseed, olive, rapeseed, and safflower oils tend to yield biodiesel with CP ≤ 0°C; our results agree with Kalligeros (2003) and Peterson (1987).

4. Conclusion:

In this study the application of ultrasound during extraction and transesterification of oil from rapeseed was evaluated. Extraction was done by two methods, batch wise extraction (conventional and ultrasonicated) and Soxhlet extraction (conventional and ultrasound). Ultrasonic technique reduced time required to extract oil. The oil composition is not affected by the use of ultrasound. The alkaline transesterification of rapeseed oil with methanol and potassium hydroxide for production of biodiesel was studied, using ultrasonication and magnetic stirring. It is concluded that a conversion of 90% was obtained for both ultrasonicated and magnetic stirred reactions after 10 min. The characterization of biodiesel obtained is in agreement with the standard biodiesel and diesel fuel worldwide.

Corresponding author
N.N. Ibiari
Chem. Eng. and Pilot Plant Dept., National Research Centre, Egypt

5. References:

1. F. Ma, M.A. Hanna, Biodiesel production; a review. Bioresour Technol. 70 (1999) 1-15. http://www.scielo.org.ar/scielo.php?pid=S032707932006000400012&script=sci_arttext DOI: 10.1016/S0960-8524(99)00025-5
2. M.K. Abu-Arabi, M.A. Allawzi, H.S. Al-Zoubi, A. Tamimi, Extraction of jojoba oil by pressing and leaching, Chem. Eng. J. 76 (2000)61-65 http://linkinghub.elsevier.com/retrieve/pii/S1385894799001199 doi:10.1016/S1385-8947(99)00119-9
3. G.L. Silva, I.S. LEE, A.D. Kinghorn, Special problems with the extraction of plants. Pp. 343-363. In Cannell, R.J.P. (ed.): Methods in Biotechnology 4. Natural Products Isolation. Humana Press, Totowa, New Jersey, USA. 1998.
4. D.A.J. Starmans, H.H. Nijhuis, Extraction of secondary metabolites from plant material: a review, Trends Food Sci. Technol. 7 (1996) 191-197.
5. C.W. Huie, A review of modern sample preparation techniques for the extraction and analysis of medicinal plants, Anal. Bioanal. Chem.373(2002)23-30. www.springerlink.com/index/6EG8J7Y2L0HWJXQB.pdf Doi:10.1007/s00216-002-1265-3
6. P. Valéria Flores, S. Jenifer, M. S. Maria Ines, A. C. Fernanda, De A. J. Rosangela, M. M. Migdalia, C. O. Eniz, C. B. Elina, Comparison of soxhlet, ultrasound-assisted and pressurized liquid extraction of terpenes, fatty acid and vitamin E from piper gaudic haudianum kunth. Journal of Chromatography A. 1105 (2006) 115-118.http://linkinghub.elsevier.com/retrieve/pii/S0021967305016122 doi:10.1016/j.chroma.2005.07.113
7. J.L. Luque-Garcia, M.D. Luque De Castro, Ultrasound – assisted soxhlet extraction an expeditive approach for solid sample treatment. application to the extraction of total fat from oleaginous seeds. Journal of Chromatography A, 1034 (2004) 237-242. http://linkinghub.elsevier.com/retrieve/pii/S0021967304001992 doi:10.1016/j.chroma.2004.02.020
8. M. Vinatoru. An over view of the ultrasonically assisted extraction of bioactive principles from herbs, Ultrasonics Sonochemistry. 8 (2001) 303-313. http://linkinghub.elsevier.com/retrieve/pii/S1350417701000712 doi:10.1016/S1350-4177(01)00071-2
9. A. Demirbas, Biodiesel fuels from vegetable oils via catalytic and non-catalytic supercritical alcohol transesterifications and other methods: a survey, Energy Conversion and Management. 44 (2003)2093-2109. http://linkinghub.elsevier.com/retrieve/pii/S0196890402002340 doi:10.1016/S0196-8904(02)00234-0
10. A. S. Ramadhas, S. Jayaraj, C. Muraleedharan, Biodiesel Production from high FFA Rubber seed Oil, Fuel. 84 (2005) 335-40. http://linkinghub.elsevier.com/retrieve/pii/S0016236104002844 doi:10.1016/j.fuel.2004.09.016
11. SV. Ghadge, H. Raheman, Process optimization for biodiesel production from mahua (Madhuca Indica) oil using response surface methodology. Bioresour Technol. 97(2006)379-384. http://linkinghub.elsevier.com/retrieve/pii/S0960852405001744 doi:10.1016/j.biortech.2005.03.014
12. H. Fukuda, A. Kondo, H. Noda, Biodiesel fuel production by transesterification of oils. J Biosci Bioeng. 92 (2001) 405-16. http://linkinghub.elsevier.com/retrieve/pii/S1389172301802887 doi:10.1016/S1389-1723(01)80288-7
13. Y. C. Sharma, B. Singh, S.N. Upadhyay, Advancements in development and characterization of biodiesel: A review, Fuel. 87

(2008) 2355-73. http://linkinghub.elsevier.com/retrieve/pii/S001623610800029X doi:10.1016/j.fuel.2008.01.014
14. B. Freedman, RO. Butterfield, EH. Pryde. J. Am Oil Chem. Soc, 61 (1986) 1375-80.
15. M. Canakci, J. V. Gerpan, Biodiesel production via acid catalysis trans. Am. Soc. Agric. Eng. 42 (1999) 1203-10.
16. M. Canakci, J.V. Gerpan, Biodiesel production from oils and fats with high free fatty acid. Am. Soc. Agric. Eng. 44 (2001) 1429–36.
17. V.B. Veljkovic, S.H. Lakicevic,O.S. Stamenkovic, Z.B. Todorovic, M.L. Lazić, Biodiesel production from tobacco oil with a high content of free fatty acids, Fuel. 85 (2006) 2671-75. http://linkinghub.elsevier.com/retrieve/pii/S001623610600144X doi:10.1016/j.fuel.2006.04.015
18. P.K. Sahoo, L.M. Das, M.K.G. Babu, S.N. Naik, Biosiesel development from high acid value polanga seed oil and performance evaluation in a CI engine. Fuel 2007; 86, 448-54. http://linkinghub.elsevier.com/retrieve/pii/S00162361060003024 doi:10.1016/j.fuel.2006.07.025
19. U.C. Shanna, B. Singh, Development of biodesel from karanja, a tree found in rural India, Fuel. 87 (2007) 1740-42. http://linkinghub.elsevier.com/retrieve/pii/S001623610700366 3 doi:10.1016/j.fuel.2007.08.001
20. DYC Leung, Y. Gvo, Transesterification of neat and used frying oils, Optimization for biodiesel production. Fuel Process Technol. 87 (2006) 883-90.
21. G.-T. Jeong, D.-H. Park, C.-H. Kang, W.-T. Lee, C.-S. Sunwoo, C.-H. Yoon et al, Production of biodiesel Fuel by transesterification of rape seed Oil. Applied Biochemistry and Biotechnology. 114 (2004)747-458. http://www.springerlink.com/index/DJ3J7216111215Q7.pdf doi:10.1385/ABAB:114:1-3:747
22. Tiwari AK. Kuman A. Raheman H. Biodiesel production from Jatropha oil (Jatropha curcas) with high free fatty acids: an optimization process. Biomass Bioenergy 2007; 31:569-75.
23. Al-Widyan MI. Al-Shyoukh AO. Experimental evaluation of the transesterification of waste palm oil into biodiesel. Bioresour Technol 2002;85:253-6.
24. C. Stavarache, M. Vinatoru, R. Nishimura, Y. Maeda, Fatty acids methyl esters from vegetable oils by means of ultrasonic energy, Ultrasonic Sonochemistry 2005, 12: 367-372. http://linkinghub.elsevier.com/retrieve/pii/S135041770400080X doi:10.1016/j.ultsonch.2004.04.001
25. K. Loren, Manufacture of cooking oils and margarine #2, 12/27/2007 http://www.karlloren.com/Diabetes/p43.htm
26. J. Lifka, B. Ondruschka, Influence of mass transfer on the production of bio- diesel, Chemical Engineering and Technology. 27 (2004) 1156-1159.
27. Hochhauser, A.M., Gasoline and Other Motor Fuels, in Kirk-Othmer, Encyclopedia of Chemical Technology, 4th edn., edited by J.I. Kroschwitz and M. Howe-Grant, John Wiley & Sons, New York, Vol. 12, 1994, pp. 341–388.
28. Kalligeros, S., F. Zannikos, S. Stournas, E. Lois, G. Anastopoulos, Ch. Teas, and F. Sakellaropoulos, An Investigation of Using Biodiesel/Marine Diesel Blends on the Performance of a Stationary Diesel Engine, Biomass Bioenergy *24:* 141–149 (2003).
29. Peterson, C.L., R.A. Korus, P.G. Mora, and J.P. Madsen, Fumigation with Propane and Transesterification Effects on Injector Cooking with Vegetable Oils, Trans. ASAE *30:*28–35 (1987).

10/30/2010