Kathmandu University
Journal of Science, Engineering and Technology
Kavita *et al*. Vol. 11, No. I, August, 2015, pp 61-68.

SHORT COMMUNICATION OPEN ACCESS

WASTE VEGETABLE OIL: A BOON FOR BIODIESEL PRODUCTION

[1]Kavita S H*, [2]Krishnamurthy V, [3]Chandra Prabha M N, [4]RoshanMakam
[1,2,4]Department of Biotechnology, PES University, 100 Ft. Ring Road, BSk III Stage, Bengaluru-560 085
[3]Department of Biotechnology, MSRIT, MSR College Road, MSR Nagar, Bengaluru,– 560 054

*Corresponding author:shkavitha@yahoo.com
Received 18 June, 2015; Revised 29 July, 2015

ABSTRACT
Edible Waste Vegetable Oils (WVO) are potential water pollutants that exert strain on community water treatment plants. They are therefore recalcitrant. Utilising them as feed stocks to convert into biodiesel by trans-esterification process is the best use to address water pollution as well as to meet the growing fuel demand. Being a bio-fuel, the pollution could be also minimal and hence this study was aimed to synthesise bio-diesel from WVO. An indigenously designed, fabricated, modular and mobile bio-diesel reactor plant was empolyed to manufacture 40 liters of biodiesel a day, from WVO feed stock by a series of reaction-optimised processes. The biodiesel so obtained was subjected to standard fuel tests and the values compared well with the prescribed corresponding ASTM standards. The cost also worked out economical, at thirty rupees per liter. The fuel was also field tested with two of its applications. Firstly, as a blend at 20 % level with commercial diesel in the fuel tank of heavy motor vehicle and secondly as a boiler oil fuel to heat up the water. The incidental by-product namely the glycerol also had commercial value.

Keywords: Bio-fuel, fried oil waste, bioreactor, modular, trans-esterification, renewable energy, diesel, glycerol

INTRODUCTION
There is an exponential demand for energy and there is a need for renewable energy source to produce fuels that cause less pollution. Biodiesel falls in this category. Biodiesel is produced using trans-esterification process from various types of oils. Different methods such as acid catalysis, base catalysis and enzyme catalysis have been adopted to produce biodiesel [1]. Waste vegetable oils from restaurants, fried potato chips, etc. are a good source of raw material for biodiesel production [8]. The impediment is that they contain free fatty acids which can lower the yield [5].
Edible vegetable oils after their usage become recalcitrant waste which becomes environmental pollutants that contaminate water and soil [7]. Making use of such waste and converting it into precious resource like biodiesel production would be therefore a novelty. Such an effort of using waste oil and subjecting it to trans-esterfication process upon optimizing the required chemical process parameters has been attempted successfully in this study.
In this study, waste vegetable oil (WVO) was obtained from various sources and pooled together. The amount of free fatty acids (FFA) was estimated to be less than 1%. Initial experiments dealt with optimising experimental process parameters. The parameters included catalyst, methanol to oil molar ratio and temperature using both acid and base catalysis [3].

Kathmandu University
Journal of Science, Engineering and Technology
Kavita et al. Vol. 11, No. I, August, 2015, pp 61-68.

Alkaline trans-esterification gave better results when compared to acid catalysis [4]. Methanol and sodium hydroxide were used in all these experiments because of their low cost. The optimized parameters were catalyst (Sodium Hydroxide = 6.25 g/L of WVO, Methanol = 25% volume by volume of WVO) and temperature = 60 ^{0}C.

MATERIALS AND METHODS

A 5 L capacity lab scale all stainless steel biodiesel plant was designed. It consisted of two reactor vessels, one for the production of biodiesel (Diameter = 25.2 cm and Height = 25.7 cm) and the other was for washing the produced biodiesel (Diameter = 23.6 cm and Height = 24.0 cm). The vessel producing biodiesel consisted of an agitator with a motor for uniform agitation of the reactants and catalyst. Both the vessels were mounted on a stand. The product vessel was heated with a heating mantle to a 60 ^{0}C temperature for the trans-esterification reaction to occur.

The following table presents the summary of activities and purposes undertaken during this study.

Table 1: Biodiesel production – Sequelae of the study

SN	Activity	Purpose
1	**Design of lab scale reactor:** Fabrication of 5 L All stainless steel reactor	• To understand trans-esterification process parameters and product testing. • To get experience for designing pilot plant
2	**Design of pilot plant:** Optimization of process parameters for pilot plant process(25L capacity)	• To scale up yield • To indigenously design a mobile Biodiesel plant
3	**Allied activities:** (a) Testing of product (b) By- product	• Product testing and comparison with ASTM prescribed standards • Glycerol as By-product • Costing process

Process Flow Diagram

A process flow diagram showing schematic representation is given below (Fig.1).

Step 1:Pre-treatment of the waste vegetable oil sample:-

The following steps outline the pre-treatment

- ***Pre-heating*** – The oil sample was heated to 50-60^{0}C. This was carried out to decrease the viscosity of oil and thus making filtration easy.
- ***Filtration***– Filtration of oil was carried out to remove the sediments (impurities) present in the oil sample. This was achieved by using a muslin cloth.
- ***Heating***– The filtered oil sample was heated to above 100^{0}C in order to remove the excess water present in the oil sample since excess water can lead to saponification.
- ***Determination of FFA%*** - Free fatty acid % was determined by titration of oil against NaOH. 10ml of oil + 10ml of iso-propanol + few drops of phenolphthalein indicator. Titration of the reaction mixture with Sodium Hydroxide was carried out till the color changed to pink and persisted for approximately 60 seconds.

Kathmandu University
Journal of Science, Engineering and Technology
Kavita et al. Vol. 11, No. I, August, 2015, pp 61-68.

Step 2: Process

Transesterification: The major component of vegetable oil is triglycerides. In this reaction, triglycerides are converted to diglyceride, monoglyceride, and finally converted to glycerol and biodiesel. When the triglycerides react with alcohol in the presence of a base catalyst, it is called “transesterification”.

The sequential steps involved in process:-

- The oil was heated to the required temperature (60^0C).
- Sodium Methoxide was prepared by mixing a known quantities of Sodium Hydroxide and Methanol
- The sodium methoxide so prepared was mixed with the oil which had reached the required temperature.
- The reaction was allowed to occur for 1 hour at the specific conditions of the temperature and pressure.
- The reaction mixture was allowed to cool to room temperature when two layers got separated: constituting Biodiesel (upper layer) and glycerol (lower layer).
- The glycerol was recovered from the bottom of the reaction vessel

Step 3: Washing

- Based on the volume of biodiesel produced $1/4^{th}$ volume of water was warmed to 45^0C and mixed with the biodiesel; stirred in a separatory funnel for 30 minutes and allowed to settle for 3-4 hours. The two layers formed were of water (lower layer) and Biodiesel (upper layer). Washing of the product was continued until the pH of the bottom layer of water reached 7.

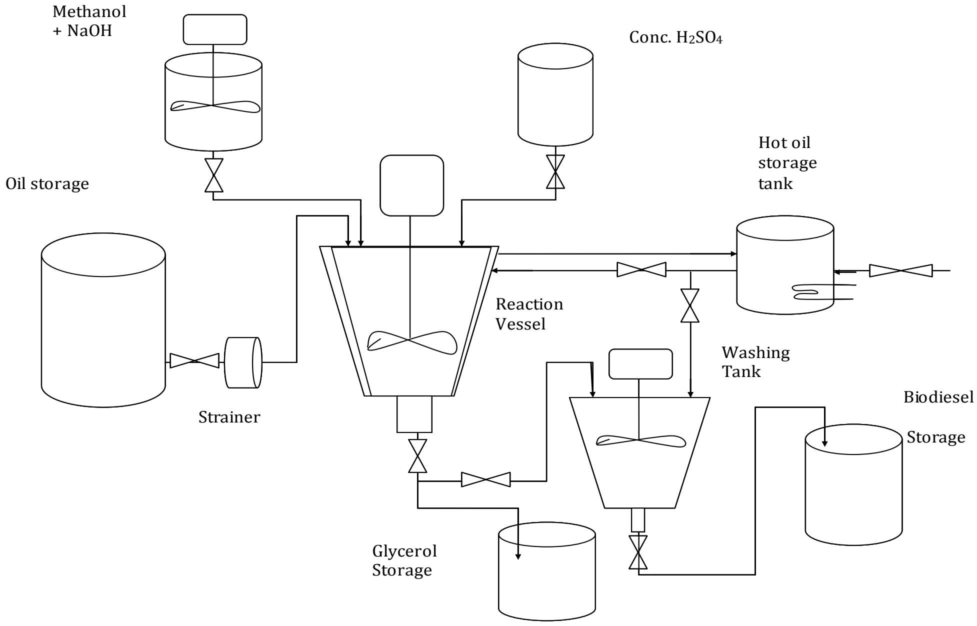

Figure 1: Schematic process diagram for production of biodiesel

Another attempt to optimize experimental parameters was done using Design Expert 8.0 software [2, 6]. . A 2^3 equal to 8 experiments using two levels were run at lab scale (200 ml of WVO) to optimize the parameters catalyst percentage, methanol to oil molar ratio and

Kavita *et al*. Vol. 11, No. I, August, 2015, pp 61-68.

temperature .The two levels were chosen such that the design space was operated within these two levels. The levels consisted of low and high values of the parameters. Sodium Hydroxide was in the range of 0.5 – 1.0%, Methanol to Oil molar ratio was between 3:1 – 7:1 and the temperature was 60 ^{0}C and 70 ^{0}C. The optimum parameters were found to be Sodium Hydroxide 0.75%, Methanol to Oil molar ratio was 5:1 and the temperature was 60^{0}C. The yield of biodiesel obtained was 99-100%. Using these optimized parameters a 25 L capacity biodiesel pilot plant was designed so that 45 L of biodiesel per day could be produced in three batches. The biodiesel production plant was built on skid, enabling mobility of the whole plant.

Optimized Pilot Plant Process

- 10 L of Waste Vegetable Oil was poured through the inlet valve and pre-heated to 105^{o}C with the vent open to let the excess moisture in the oil(if any) to evaporate. It was then cooled to the reaction temperature, 60^{o}C, and the vent was closed not to allow extraneous air.
- 75 grams of Sodium Hydroxide was dissolved in 2.18 L of Methanol to prepare Sodium Methoxide which is the catalyst.
- The reactants were poured into the pilot plant reactor through the inlet valve and it was closed so as to make the vessel air-tight again. This ensured evaporation of Methanol out of reaction vessel.
- The reaction was carried out at 60^{o}C for 1 hour maintaining stirrer speed of 300 rpm.
- At the end of 3-4 hours the glycerol formed settled at the bottom of the vessel which was collected from outlet valve.
- The Biodiesel obtained was subjected to washing; 2.5 L of distilled water was taken for washing the Biodiesel sample, warmed to 55^{o}C for 20 minutes with a stirrer speed of 300 rpm.
- After 20 minutes, the mixture of Biodiesel formed and water was made to stand for 2 hours when two distinct layers are formed - the lower of water and the upper layer of Biodiesel.
- Water at the bottom was drained out from the outlet valve of the separating unit and the Biodiesel was retained in it.
- The pH of the water collected from the bottom of the reaction vessel was checked.
- The washing was continued until the pH of washed water reached 7 and then biodiesel obtained was considered as the end product and stored.

Kathmandu University
Journal of Science, Engineering and Technology
Kavita *et al*. Vol. 11, No. I, August, 2015, pp 61-68.

Indigenously designed and fabricated Biodiesel plant with specifications are provided in the Figure 2 and Table3.

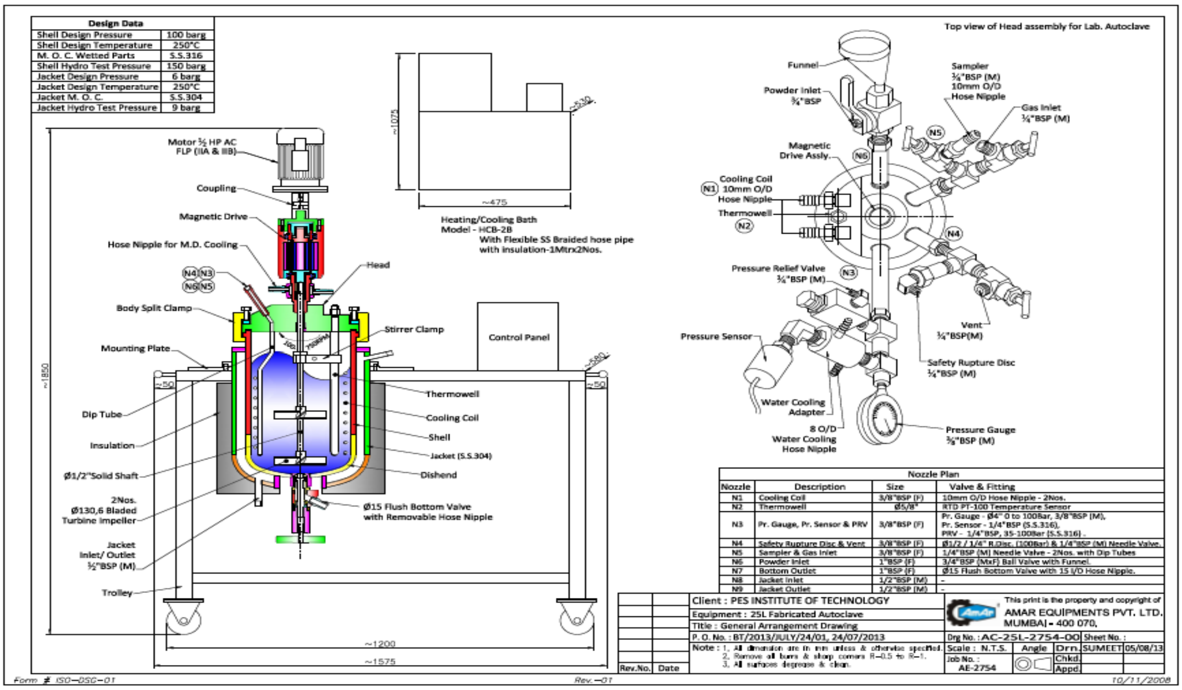

Figure 2: Engineering drawing of the reactor for trans-esterification process to produce biodiesel

Fig 3: Pilot plant for production of biodiesel from Waste Vegetable Oil

Kathmandu University
Journal of Science, Engineering and Technology
Kavita *et al.* Vol. 11, No. I, August, 2015, pp 61-68.

Table 2: Technical Specification for the SKID Mounted SS 316 Reactor of 25 L Capacity

Reactor Part	**Parameter**	**Specification**
Reactor	Nominal Volume (lt)	25 Lt. Nominal Volume
	L/D Ratio	1.2
	Nozzles	1/2" 4 Nos. on the Top Dish; 1/2" 2 Nos. on the Reactor Shell.
	Reflux Cooler	Double Pipe Heat Exchanger Type; 0.25 m^2 Area
Agitation	Drive	AC Geared Motor
		Top drive(mechanical seal driving)
	Motor Power & Drive	0.5 hP with Variable Frequency Drive, with RPM indication
	Range	50 - 500 rpm
	Impellers	Rushton turbine / PBT Impeller, Foam breaker; All (SS316L)
Sealing	Shaft	Zero leakage Magnetic drive (Maintenance free)
Materials & Finish	Vessel	316L stainless steel ; polished mechanicaly, GMP
	Piping	316L stainless steel ; polished mechanicaly, GMP
	Nozzles	316L stainless steel ; polished mechanicaly, GMP
	Condenser	0.022 m2 SS-316 Reflux / take off condensor
	O-rings/Gaskets	Teflon
	Pressure Safety relief	(1/2”) of MOC SS- 316 (3 - 4 Bar)
Jacket Heating & Cooling	Range(ºC)	Room Temperature to 150
	Sensor	RTD(Pt-100)
	Control	Microprocessor controlled heating and cooling Systems through hot flow of oil in the Jacket
Aeration	Sparger	Ring type sparger(SS 316L)
	Intel Filter	The cartridge type sterilizable pleated type 0.2 absolute filter specifications
	Out Filter	The same as intel filter specification
	Control Range	Manual Control by Rotameter
Pressure Control	Full Vacuum to 6 bar gauge pressure Manual using a back control valve (optional item)	
Sensors	pH	
	DO	
	RTD(Pt-100)	
	Pressure Transmitter	
	Agitation Speed	
Power Supply	220V 50/60Hz, Single phase	
Data Storage	pH, DO, Pressure, Temperature and Agitation speed	

Kathmandu University
Journal of Science, Engineering and Technology
Kavita *et al*. Vol. 11, No. I, August, 2015, pp 61-68.

RESULTS AND DISCUSSION

Two test runs were conducted using 10 & 15 L of WVO as stock source. The catalyst in % was 0.75 for both the runs; temperature maintained for reaction in both the runs was 60°C and a molar ratio of 1:5 was maintained. The yield obtained was 99.6 and 99.7 % respectively for the two runs.

The Biodiesel thus obtained was then tested for the following parameters:- Flash Point, Fire point, Pour point, Cloud point Specific gravity, Copper Strip Corrosion test and Kinematic Viscosity and is listed in Table 3 below:

Glycerol by-product: Glycerol was produced as a by-product in the main trans-esterification reaction. Approximately 6 L of glycerol was produced per day, which contained amounts of biodiesel, unreacted methanol, sodium hydroxide, and soap, (which are not included in the mass balance). Purification of glycerol involves a neutralization step, and the separation of excess methanol, and salts through a washing step. Purified glycerol finds variety usage including medicinal and the cost of of glycerol is approximately 300 Rs/ Kilogram.

Table 3: Properties of Biodiesel

SN	Fuel property	Biodiesel sample	ASTM D-6751
1	Kinematic viscosity (mm^2/s, at40^0C)	4.45	1.9 -6.0
2	Specific gravity (kg/L,at RT)	0.882	0.075-0.840
3	Flash Point (^0C)	175	>130
4	Fire Point (^0C)	175	> 130
5	Cloud Point(^0C)	12	-3 to 12
6	Pour Point(^0C)	-2	-15 to 10
7	Copper strip corrosion test 3 h at 50°C	No corrosion	0.02

The fuel properties studied are well compared with prescribed ASTM standards. Majority of the values of parameters tested fell within the standard limits. This indicates that the fuel quality check is successful. The fuel was also put to test by using it in motor-vehicle fuel tank (college buses) as a blend (20%) with the commercial diesel. The feedback from the vehicle experts and the experience has been good. The biodiesel has been used as water heating boiler fuel that exists in the boys hostel of the University. The heating capacity was proved to be good. Thus there were no issues of any sort with both these utilities for the synthesised biodiesel.

CONCLUSIONS

The intent of the study was to manage liquid waste oil that could be a part of water pollution. The utilisation of waste cooking oil to synthesis of biodiesel that is equivalent to commercially manufactured from fossil sources by optimised reaction process in a custom-built reactor is the novelty of the study. A well thought, indigenously designed, mobile biodiesel plant with a production capacity of 45 L/day has been achieved. The production cost of Biodiesel so produced worked out to be thirty rupees (INR 30/-) which is inclusive of the cost of all the raw materials; hence the process is economical. The quality of the Biodiesel has been found to be commensurate with that prescribed by ASTM standards. The synthesised biodiesel has been used successfully as a blend with fossil derived diesel to run vehicles as well as use as a heating fuel in water boilers. Results with both the usages have been good. Glycerol, obtained as by-product is proved to be an incidental by product that has an economic benefit. The exercise paved way for a small enterprise of manufacture of biodiesel and also addressed the issue of water pollution by saving treatment of water from domestic oil pollution.

ACKNOWLEDGEMENT

The financial support to fabricate the bioreactor was extended by Professor Jawahar, the CEO of PES institutions and authors are ever grateful for this support. The authors gratefully acknowledge the Dr, KNB Murthy, Vice-Chancellor of PES University for his encouragement and support for the project. Authors also acknowledge students and staff members of biotechnology department of PES Institute of Technology and PES University.

REFERENCES

[1] Refaat, N. K. Attia, H. A. Sibak, S. T. El Sheltawy, and G.I. ElDiwani, Production optimization and quality assessment of biodiesel from waste vegetable oil, International Journal of Environmental Science and Technology, 5(2008) 75.

[2] De Poola, M.G., Ricca, E., Calabro,V., Curcio and S., Iorio, G , Factorial analysis of transesterification reaction of waste oil for biodiesel ,BioresourceTechnology, 100 (2009) 5126.

[3] M. S. Hossain, A. N. Boyce, A. Salleh, and S. Chandran, Impacts of alcohol type, ratio and stirring time on the biodiesel production from waste canola oil, African Journal of Agricultural Research, 5(2010)1851.

[4] M. Ahmad, S. Ahmed and F. U. Hassan, Base catalyzed transesterification of sunflower oil biodiesel, African Journal of Biotechnology, 9(2010)8630.

[5] M.Mathiyazhagan, A. Ganapathi, B. Jaganath, N. Renganayaki, And N. Sasireka, Production of biodiesel from non-edible plant oils having high FFA content, International Journal of Chemicaland Environmental Engineering, 2(2011)119.

[6] Yusra Safa and Haq Nawaz Bhalti, Adsorptive removal of direct textile dyes by low cost agricultural waste: Application of factorial design analysis, Chemical Eng. Journal, 2(2011)35.

[7] Mukesh A Mane, Karanja oil as an Alternative Fuel for Direct Injection CI Engine, International Journal of Science and Research, 2(2013) 2319.

[8] Eze, S. O., Ngadi, M. O., Alakali, J. S. and Odinaka, C. J, Quality Assessment of Biodiesel Produced From After Fry Waste Palm Kernel Oil (PKO), European Journal of Natural and Applied Sciences, 1(2013) 38.