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Extraction of Tobacco (Nicotiana tabacum L.) seed oil and Biodiesel preparation through two stage transesterification

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ABSTRACT

Biodiesel derived from vegetable oils is found to be one of the promising source of alternative to conventional fossil fuels. Extraction of oil from tobacco seed and production of biodiesel in a two stage trans-esterification process from tobacco seed oil is experimentally investigated. The oil extraction procedure employed in the Soxhlet apparatus, where n-hexane is used as a solvent to separate oil from cell wall membrane of crushed tobacco seeds at 70°C. Acid catalysed and base catalysed trans-esterification was employed to reduce the free fatty acid content and to convert the treated oil into biodiesel respectively. The yield of biodiesel were analysed with NaOH and KOH as catalyst and also by varying the molar ratio between 1:4 and 1:8. The optimum yield of biodiesel was noticed at molar ratio 1:6, reaction temperature 60°C and reaction time 100 minutes with NaOH as catalyst.

Keywords: Biodiesel, Trans-esterification, Soxhlet apparatus, Sulphuric acid

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INTRODUCTION

Increased energy crisis, rapid depletion of petroleum reserves and faster rate of environmental pollution created a huge necessity for the researchers to identify an alternative source of fuel for petro-diesel. Biodiesels can be derived from vegetable sources, animal fat, waste inorganic materials (waste tire, plastic and pyrolytic oil) and micro- macro organisms. Non edible vegetable oils like Neem, Jatropha, Pongamia, Karanja and many more yield biodiesel through single and two stage trans-esterification process for its use in an internal combustion engine [6]. As an alternative to petroleum diesel, biodiesels should be comparable with its physio chemical properties, cost and reliability to survive in the world market. India being second largest tobacco cultivator in the world about 0.25% of total cultivable land is used for tobacco cultivation. Mainly in areas like Jeelugumilli, Kandukuru, Guntur, Kalavacherla, Rajamundry in Andhra Pradesh, Humsur in Karnataka, Vedasandur in Tamilnadu and Dinhata in West Bengal. Tobacco seed oil is one such alternative to petroleum diesel which has comparable properties with petro diesel. Since the free fatty acid content and the acid value of tobacco seed oil is on the higher side, two stage trans-esterification process is to be applied for transforming into biodiesel [2-4].

Veljkovic et al. presented the oil extraction procedure from tobacco seeds using n-hexane and petroleum ether as solvents at various temperatures. Soxhlet extraction procedure was employed which yielded 31.1 grams of tobacco seed oil using n-hexane solvent whereas petroleum ether solvent extracted only about 23.5gms of tobacco seed oil from 100grams of dry tobacco seed. They also analysed that linoleic acid was the major constituent of tobacco seed oil with high acid value [18]. He also experimented the production of biodiesel from tobacco seed oil using two stage trans-esterification process. Since the free fatty acid content of tobacco seed oil was greater than 35%, they have used acid catalysed esterification followed by base catalysed esterification. The reaction yielded a maximum of 91% biodiesel at a reaction time of 30 minutes. They also tested and compared the properties of tobacco seed biodiesel and found to be within ASTM standards [19].

Usta et al. reported on the improvement of the physio chemical properties of tobacco seed oil with additives. They have analysed on the improvement of oxidation stability, iodine number and cold flow improvements with additives like tert-butylhydroquinone, butylated hydroxytoluene, propyl gallate, pyrogallol, α-tocopherol, butylated hydroxyanisole, ethylene – vinyl acetate copolymer and octadecene – 1 – maleic anhydride copolymer [17]. They analysed the effect of tobacco seed biodiesel in a turbo charged indirect injection diesel engine. Soxhlet extraction apparatus was used for the extraction of tobacco seed oil with diethyl ether as extraction solvent in a Buchi R114 rotary vacuum evaporator. The extraction efficiency was found to be 38% on weight basis. The trans-esterification at a molar ratio of 6:1, 7.5gms of NaOH catalyst at 55°C yielded 86% of tobacco seed biodiesel. The properties were found to be within DINEN14214 standards [16].

In the presence study, tobacco seed oil was processed from grounded tobacco seeds obtained from CTRI, Vedasandur, Tamil Nadu. Soxhlet extraction apparatus was employed for extraction of oil using n-hexane as a solvent at 70-80°C. Acid catalysed trans-esterification followed by base catalysed trans-esterification was carried out to convert the tobacco seed oil into its biodiesel. An optimisation study was also conducted by varying reaction temperature, catalyst concentration and molar ratio to analyse the yield of biodiesel.

MATERIALS AND METHODS

5kg of hybrid tobacco (Nicotiana tabacum L.) seed (Abirami variety) was purchased from Central Tobacco Research Institute, Vedasandur, Dindugal District, Tamil Nadu, India. The seeds were placed in sunlight for 48hours for drying and removal of excess moisture. The dried seeds were powdered in a grinding mill for 5 minutes at 2500rpm. By this process the cell wall membrane of tobacco seeds were broken and then subjected to oil extraction procedure in a soxhlet apparatus. It consists of three units namely heating element, extraction unit and condensation unit. 100 gms of grounded tobacco seeds were filled in the thimble and then placed in the extraction unit. The round bottom flask was filled with 500ml of n-hexane and placed on the heating mantle. Upon heating upto 70°C n-hexane vaporises and occupies the condensation unit, where the phase change of n-hexane from gaseous to liquid takes place and flows back to the round bottom flask through the thimble. Thereby due to the reaction of n-hexane and crushed tobacco seeds the lipid content was washed and collected in the round bottom flask. This procedure is repeated for 25 times with 3 minutes/cycle which extracted about 96% of tobacco seed oil. The separation of n-hexane and tobacco seed oil was carried out using a rotary evaporator at 85°C, in which 95% of n-hexane was recovered. By this process 1.8litres of tobacco seed oil was extracted [8-13].

Due to higher acid value and free fatty acid content two stage trans-esterification process was found to be suitable for transforming tobacco seed oil into biodiesel. 1000 ml of tobacco seed oil was heated up to 70°C in a three neck round bottom flask as pre-treatment. 2% of conc. Sulphuric acid by weight of tobacco seed oil was mixed with 200ml of methanol in a conical flask and then poured into pre-treated tobacco seed oil. The mixture was maintained at 60°C with 450rpm using a magnetic stirrer for 2 hours. The reaction was continuously monitored with Gas Chromatography technique for its reduction in free fatty acid content. By this process the free fatty acid content was brought down to 1.2% which is favourable to undergo the base catalysed esterification. The treated oil was subjected to washing with distilled water and drying which is followed by base catalyst esterification. 1000ml of pre-treated oil was poured in a beaker and maintained at 100°C to remove excess moisture. 200ml of methanol was mixed with catalyst (NaOH & KOH) to form methoxide solutions and then poured into the beaker to initiate the catalyst based trans-esterification process at 450 rpm and 60°C. After 90 minutes of reaction time the mixture was allowed to cool and settle down with glycerol for 12hours. During which a layer separation was seen differentiating glycerol and biodiesel. The biodiesel and glycerol were separated by gravity separation process and washed with distilled water for 5 times. The obtained biodiesel was heated upto 110°C to remove excess moisture [7-15].

RESULT AND DISCUSSIONS

The Figure 1. shows the comparison of the yield of biodiesel and reaction time with 60°C, 70°C and 80°C of reaction temperature. 500ml of n-hexane was heated in the round bottom flask which vaporises and occupies the extraction chamber in the soxhlet apparatus. The thimble containing the grounded tobacco seeds reacts with the hexane expelling oil which was collected back in the round bottom flask.

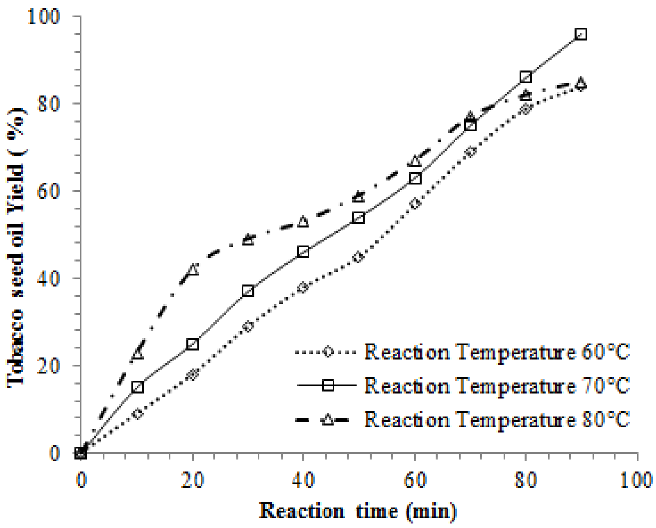

Figure 1. Effect of reaction time on the yield of tobacco seed oil at 60°C, 70°C and 80°C

At reaction temp 60°C the process of the oil extraction was slower as indicated in the figure with a maximum yield of 82% at 90 minutes reaction time. When the temperature is increased upto 70°C the reaction process was found to be better yielding 96% of tobacco seed oil. Further increasing temperature upto 80°C exhibited an improvement on the yield of tobacco seed oil upto 50minutes of reaction time but slowly reduced when the reaction time was extended upto 90minutes, which may be due to vaporisation of tobacco seed oil. The reaction time was kept constant upto 90 minutes throughout the experimentation beyond which no further improvement was noticed.

The Figure 2A and 2B shows the effect of NaOH and KOH as catalyst in the yield of tobacco seed biodiesel at reaction temp of 50°C, 60°C and 65°C. In Figure 2A NaOH is used as catalyst with concentrations between 0.3 to 0.9 grams/100ml at reaction temperature 50°C the effectiveness of catalyst in converting the tobacco seed oil into tobacco seed biodiesel was slower with a maximum of 85% was seen at 0.7grams/100ml of oil. With an increase in temperature upto 60°C the conversion rate was even better with a maximum and optimum temperature 92% with the same catalyst concentration. Further increase in temp upto 65°C exhibited a sharp reduction in the maximum yield of biodiesel by 8% to 10% which may be due to vaporisation of methanol at higher temperature.

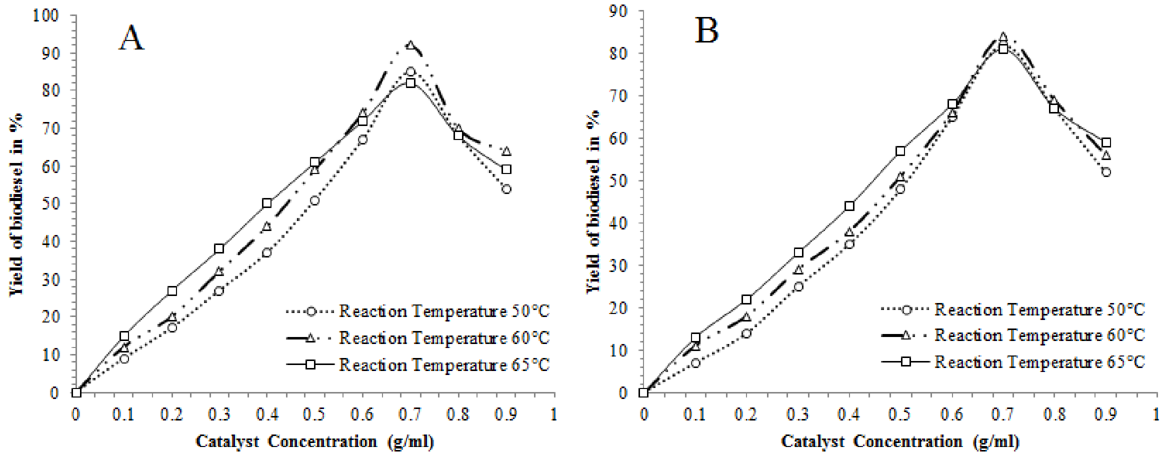

Figure 2. Effect of NaOH (A) and KOH (B) concentration on the yield of Tobacco seed biodiesel

At all reaction temperature increase in catalyst concentration beyond 0.7grams/100ml showed a drastic reduction in the yield of biodiesel, due to sludge formation in the reaction chamber. Figure 2B exhibits the effect of KOH as catalyst on the yield of biodiesel with similar reaction environments. The optimum yield of biodiesel was found to be between 81% and 84% with KOH concentration of 0.6 to 0.7gm/100ml at reaction temperature of 60°C.

Figure 3. shows the effect of molar ratio on the yield of tobacco seed biodiesel at 60°C reaction temperature. The molar ratio was varied between 1:4 and 1:8 in which the optimum yield of biodiesel was noticed at 1:6, oil to methanol ratio. At lower molar ratio the yield of biodiesel was found to be 75% at reaction time 100minutes.

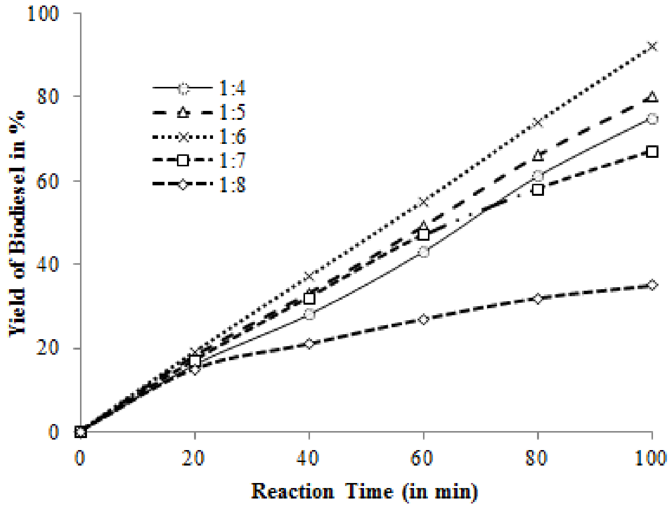

Figure 3. Effect of molar ratio on the yield of Tobacco seed biodiesel.

The yield of biodiesel was noticed to increase with an increase in concentration of methanol (increase in molar ratio) upto 1:6 were the maximum yield of biodiesel was found to be 92% at reaction temperature 60°C at 100minutes reaction time. Further increase in molar ratio upto 1:8 exhibited negative effect on the yield of biodiesel with a trans-esterification efficiency of 35% which may be due to addition of excess methanol

[6]. The reaction temperature was maintained at 60°C beyond which no favourable increase in the yield of biodiesel was noticed and the methanol also vaporises beyond 64.7°C.

Table 1. Comparison of Physio-Chemical properties of Tobacco seed oil, Tobacco seed biodiesel, with diesel and biodiesel from Karanja, Cotton and Soyabean

Properties / Biodiesel	Tobacco seed oil	Tobacco seed biodiesel	Karanja seed biodiesel	Cotton seed biodiesel	Soyabean biodiesel	Diesel
Density (kg/m^3)	943.6	887.8	881	875	882	814
Kinematic viscosity	62.8	3.5	4.4	5.9	4.09	4.3
Calorific value (kJ/kg)	38540	39811	37595	40585	39775	43350
Iodine value	135	109.8	87	89	127	NA
Saponification value	193	172	186	191	188	NA
Acid value	3.35	0.45	0.42	0.98	1.1	0.36
Cetane number	46	52.1	55	44.7	36	47
Flash point (°C)	289	212	456	385	445	71

Comparison of the physio chemical properties of tobacco seed oil and tobacco seed oil methyl ester with diesel and biodiesel from Karanja seed, Cotton seed and Soyabean is discussed in Table 1. It can be noticed that the kinematic viscosity of tobacco seed oil was brought down from 62.8mm^2/s to 3.5mm^2/s through two stage trans-esterification process. The density of tobacco seed biodiesel was found to be 887.8 kg/m^3 on comparison with diesel as 814kg/m^3 and 881 kg/m^3, 875 kg/m^3 and 882kg/m^3 for KSB,CSB and SSB respectively. The calorific value of tobacco seed biodiesel was found very similar to SSB which was increased by 3-4% due to trans-esterification. The iodine value of tobacco seed biodiesel was noticed to be 109.8 which was lying intermediate between 87 and 127 for KSB and SSB respectively. Saponification value seems to be lowest at 172 on comparison with other biodiesels. The tobacco seed biodiesel have better cetane number (52.1) which may lead to better combustion [1,5,20].

CONCLUSION

In the present experimental investigation, oil is extracted from tobacco seed and biodiesel is prepared using two stage trans-esterification. The experimental results are discussed below,

- Soxhlet apparatus was used to extract oil from crushed tobacco seed with n-hexane as solvent at optimal reaction time of 90minutes and reaction temperature of 70°C.
- The acid catalysed esterification using concentrated sulphuric acid and methanol reduced the FFA content from 14% to 1.2%
- The effect of NaOH & KOH were analysed on the preparation of biodiesel as catalyst. 0.7grams/100ml of NaOH and 0.9grams/100ml KOH were investigated experimentally with 1:5 v/v ratio methanol which resulted in a maximum 92% of biodiesel yield, obtained with NaOH as catalyst.
- Molar ratio of between 1:4 and 1:8 of oil to methanol were investigated and molar ratio 1:6 proved to be yielding 92% of tobacco biodiesel at 60°C reaction temperature and reaction time of 100 minutes
- The physio chemical property of tobacco biodiesel was found to be comparable with diesel and other biodiesels and was within ASTM standards.

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