



Soxhlet Extraction and FTIR Characterization of Biodiesel from Oil Seeds

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Abstract: In the present work Soyabean, groundnut, cotton, brown flaxseed, coriander and clove seed oils were extracted using soxhlet extractor. Characterization of these extracted oils were done on FTIR spectrophotometer. The extracted oils were used for the production of biodiesel by trans esterification process. The synthesized biodiesels were also characterized using FTIR spectroscopy.

Keywords: Soyabean oil, Groundnut oil, Cotton seed oil, Brown flaxseed oil, Coriander, Clove seed oil, biodiesel, Soxhlet extraction.

Introduction:

Extraction and evaluation of sandalwood tree seed (santalum) oils were studied by P.N. Okolie et al [1]. Extraction and characterization of vegetable oils from cherry seed by different extraction processes was studied by Maria Cristina Straccia et al [2]. Isolation, thermal efficiency, spectral characterization and microbial evaluation studies on Indian rubber (*hevea brasiliensis*) seed oil was studied by Sundaram. Arvind Narayan et al [3] reported that the fossil fuels emit greenhouse gases that cause wide harm to the environment and human health. The need for alternative fuels is rising and biodiesel is a gifted option, as it is biodegradable, less pollutant, and derives from natural and renewable feed stock. Thermal properties of biodiesel were studied by F. A. L. Machado et al [4]. Extraction and analysis of *Jatropha curcas* L. seed oil was studied by Shivani Patel et al [5]. Enzyme assisted aqueous extraction of peanut oil was studied by Sharma et al [6]. Gupta et al reported the extraction of oil from *jatropha curcas* L. seed Kernels by combination of aqueous enzymatic oil extraction and ultrasonication study [7]. Siti Machmudah et al [8] reported that the SC-CO₂ extraction cannot completely extract oil from nut seeds compared with soxhlet extraction using hexane.

Physicochemical properties and bioactive compounds of seed oils were studied by K.L. Nyam et al [9]. S. Kittiphommand and S. Sutasinee [10] were studied the mango seed kernel oil and its physicochemical properties using soxhlet extraction with petroleum ether, ethanol and hexane. M.Z. Kyari [11] also studied the extraction and characterization of seed oils. Study of *moringa oleifera* oil extraction and its influence in primary coagulant activity for drinking water treatment was studied by B. García-Fayos et al [12]. Microwave – assisted extraction of castor oil was studied by L. Mgodu et al [13]. Edison Muzenda et al [14] optimized the process parameters for castor oil production. Seung-Koo Lee [15] reported the methods for percent oil analysis of avocado fruit. Luis-Felipe Gutiérrez et al [16] reported the effects of drying method on the extraction yields and quality of oils from quebec sea buckthorn seeds and pulp. Nwobi Be et al [17] studied extraction and qualitative assessment of African sweet orange seed oil. J. T. Nwabanne [18] studied kinetics and thermodynamics study of oil extraction from fluted pumpkin seed. Weizhuo Tang et al [19] reported fast extraction of bioactive fatty acids from the perilla seeds by smash tissue extraction. R. Cardoso





de Oliveira et al [20] have extracted passion fruit seed oil using supercritical CO₂. Klára Szentmihályi et al [21] reported Rose hip (*Rosa canina L.*) oil obtained from waste hip seeds by various extraction methods. M.D. Luque de Castro et al [22] also worked on Soxhlet extraction process. Due to the potential exhausting and increasing price of petroleum together with environment concerns caused by the combustion of fossil fuels, the need for alternative fuels has gained much attention [23–25]. Biodiesel (ethyl esters or fatty acids methyl ester) , can be derived from either the trans esterification of triglycerides (the main component of vegetable oils or animal fats) or the esterification of free fatty acid (FFA) with a short chain alcohol (mainly methanol). It has become popular as a possible alternative to fossil fuels. The main advantage of this fuel are that its properties and performance are similar to conventional diesel fuels [26,27].

Waste cooking oils, soap stock and non-edible oils, which are cheaply available, are attractive starting materials for biodiesel [28,29]. Currently, most commercial processes used for biodiesel synthesis employ a homogeneous catalyst, such as NaOH or H₂SO₄ [30–32]. Georgogianni et al. [31] reported the production of biodiesel from the soybean frying oil using 2% NaOH as catalyst.

Literature survey reveals that the vegetable oils were used for the production of biodiesel. So, in the present work Soyabean , groundnut, cotton, brown flaxseed , coriander and clove seed oils were extracted using soxhlet extractor. These extracted oils were characterized by using FTIR spectrophotometer. The extracted oils were used for the production of biodiesel by trans esterification process.

Material and methods:

Operation of soxhlet extractor: The soxhlet extractions were performed using distilled water, petroleum ether, hexane and ethanol. The solvents chosen for present study are normally used to extract oil from seeds. 150 mL of solvent was poured into a round-bottom flask and 10 g seeds were added. The flask was placed inside the heating mantle. Extractor was connected to the flask and then the condenser was connected to the extractor as shown in figure 1.



Fig. 1 : Soxhlet Extractor

The solvent was boiled and evaporated. The vapour was condensed and dropped into the extractor. The solvent dripped back into the round-bottom flask. This evaporation-condensation process continued for 3 to 6 hours. Then apparatus was allowed to cool. The cold solvent-seed oil mixture was transferred into a beaker and oven heated at 70°C to remove solvent. Soxhlet extraction of oil from brown flax, cloves, coriander, cotton, soyabean and groundnut seeds was carried out.





Determination of percentage of oil content of seeds: 30g of the sample and 150 mL n- hexane were poured into the round bottom flask. The apparatus was heated at 60° C for 3 hours using Soxhlet apparatus. The experiment was repeated for 35, 40 and 50 g weights of the sample. Finally the solvent was distilled and the percentage of oil extracted was determined.

Determination of moisture contents of the seeds: 40g of the cleaned sample was weighed and dried in an oven at 80°C for 7 hrs and the weight was taken after every 2 hrs. The procedure was repeated many times until a constant weight was obtained. After each 2 hours, the sample was taken out from the oven and placed in the desiccator for 30 minutes to cool. It was then taken out and re-weighed. The moisture percentage in the seed were calculated by the formula,

$$\% \text{Moisture} = \frac{100 (W_1 - W_2)}{W_2} \%$$

Where W_1 = Original weight of the sample before drying;

W_2 = Weight of the sample after drying.

Determination of free fatty acid value: 25 ml of diethyl ether and 25 ml of ethanol were mixed in a 250 ml beaker. The mixture was added to 10 g of oil in a 250 ml conical flask and few drops of phenolphthalein were added to it. Then this mixture was titrated with 0.1M NaOH to the end point. Free fatty Acid (FFA) was calculated by the formula, $\text{FFA} = \frac{V_1}{W_0} \times 2.82 \times 100$

where, 100 ml of 0.1 M NaOH = 2.83 g of free fatty acid, W_0 = sample weight; then acidvalue = $\text{FFA} \times 2$

Synthesis of Biodeisel from Brown flax seed oil: 14 ml of pure methanol was taken in 50 mL Erlenmeyer flask having a stirrer in it. Flask was placed on a stir plate and speed was set at a rate where it doesn't splash but stirvigorously. 0.5 g of NaOH was added slowly. Stirring was continued until the NaOH being completely dissolved and sodium methoxide formed. 60 ml of pure vegetable oil was taken in a beaker and warmed to 50°C. Then sodium methoxide was slowly added to the warmed vegetable oil. Care was taken that no residual NaOH gets into the oil. Then reaction mixture was stirred for 20 minutes. The solution was transferred to a separatory funnel. It was allowed to settle for 20 minutes, the glycerol layer settled in the bottom and biodiesel layer floated in the top. Once glycerol has stopped forming, the glycerol was drained into a small graduated cylinder using the stopcock. Biodiesel layer was drained from the separatory funnel and the volume was measured. The above procedure was also repeated for the other vegetable oils viz. groundnut, soyabean, cotton seed oils.

Results and discussion:

In the present study, the extraction of oils from brown flax seeds, groundnuts, cotton seeds, cloves, soyabean and coriander seeds was carried out by using Soxhlet extractor. Different solvents for different seeds have been used such as water (Brown flax seed), ethanol (clove), petroleum ether (ground nut, coriander seeds), n-hexane (soyabean, cotton seeds). The advantage of Soxhlet extraction is that instead of different portions of warm solvent being passed through the sample containing the oil, just one batch of solvent is recycled. After the process of the extraction the solvent is removed.

Table 1. The physico-chemical properties of extracted oils:

Properties	Brownflax	Soyabean	Groundnut	Cotton
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	seeds	seeds	seeds	seeds
% of oil	18	20	15	15
Moisture content(%)	10	18	20	21
Free fatty acid value (mgKOH/g)	2.010	4.091	3.106	5.25
Peroxide value (mEq/kg)	6.06	7.09	7.9	8.25
Refractive index	0.382	0.339	1.462	1.464

Table 1 shows the physico-chemical properties of extracted oils. The % yield of oil is greater in case of soyabean and brown flax seeds as compared to the groundnut and cotton seeds. The % of free fatty acid is greater in cotton seed oil and soyabean oil as compared to the other oils. Peroxide value of cotton seed oil, groundnut oil and soyabean oil were found to be greater as compared to the rest of the oils. FTIR technique is used to identify the frequency peaks with respect to the % transmittance for confirmation of oil and biodiesel.

Cotton seed oil: Figure 2 shows the FTIR spectrum of cotton seed oil. The spectrum was characterized with asymmetric and symmetric strong stretching vibrations of carboxyl group at 2852.98 cm^{-1} and aldehyde, ketones (C=O) group along with carboxyl group at 1743.44 cm^{-1} . C-O group combined with carboxylic group at 1159.35 cm^{-1} . C-H strongly stretched at 2922.13 , 2852.98 and 1463.05 cm^{-1} . Figure 3 shows the FTIR spectrum of biodiesel prepared from cotton seed oil. The spectrum was characterized with asymmetric and symmetric strong stretching vibrations of carboxyl group in the range $3000\text{-}2500\text{ cm}^{-1}$. Aldehyde, ketones (C=O) group along with carboxylic group, ester group strongly stretched found at 1741.52 cm^{-1} . C-O group combined with carboxylic group, ester group strongly stretched at 1168.92 cm^{-1} ($1260\text{-}1000\text{ cm}^{-1}$). Alkanes (C-H) strongly stretched at $1436.01\text{-}1168.92\text{ cm}^{-1}$.

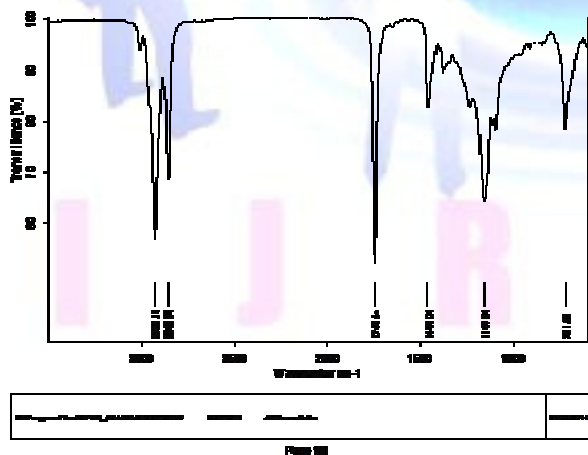


Fig.2: FTIR spectrum of cotton seed oil



Fig.3: FTIR spectrum of biodiesel prepared from cotton seed oil.

Groundnut oil: Figure 4 shows the FTIR spectrum of ground nut oil. The spectrum was characterized with asymmetric and symmetric strong stretching vibrations of carboxyl group at 2852.82 cm^{-1} and aldehyde, ketones (C=O) group along with carboxyl group at 1743.55 cm^{-1} . C-O group combined with carboxylic group at 1159.45 cm^{-1} . C-H strongly stretched at 2922.01 , 2852.82 and 1462.85 cm^{-1} . Figure 5 shows the FTIR spectrum of biodiesel prepared from groundnut oil. The spectrum was characterized with asymmetric and symmetric strong stretching vibrations of

carboxyl group in the range 3000-2500 cm^{-1} , along with the O-H stretching of the hydroxyl bonded with alcohol at 3600-3200 cm^{-1} (broad). Aldehyde, ketones (C=O) group along with carboxylic group, ester group strongly stretched at 1742.94 cm^{-1} . C-O group combined with carboxylic group, ester group strongly stretched at 1164.68 cm^{-1} (1260-1000 cm^{-1}). Alkanes (C-H) strongly stretched at 1462.06-1164.68 cm^{-1} .

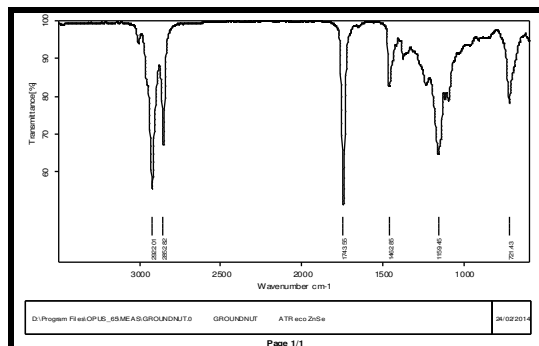


Fig.4: FTIR spectrum of groundnut seed oil

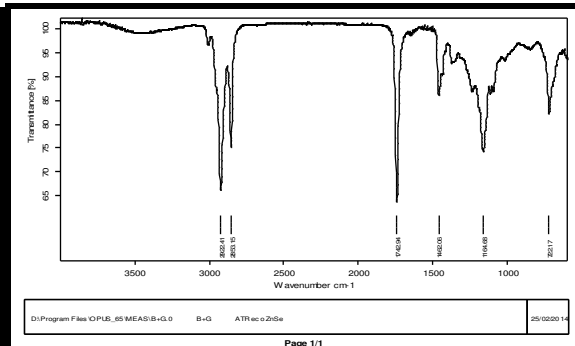


Fig.5: FTIR spectrum of biodiesel prepared from groundnut seed oil

Soyabean oil: Figure 6 shows the FTIR spectrum of soyabean oil. The spectrum was characterized with asymmetric and symmetric strong stretching vibrations of carboxyl group at 2853.29 cm^{-1} and Aldehyde, ketones (C=O) group along with carboxyl group at 1743.34 cm^{-1} . C-O group combined with carboxylic group at 1159.57 cm^{-1} . C-H strongly stretched at 2922.72, 2853.29 and 1462.33 cm^{-1} . Figure 7 shows the FTIR spectrum of biodiesel prepared from soyabean oil. The spectrum was characterized with asymmetric and symmetric strong stretching vibrations of carboxyl group in the range 3000-2500 cm^{-1} , along with the O-H stretching of the hydroxyl bonded with alcohol at 3600-3200 cm^{-1} (broad). Aldehyde, ketones (C=O) group along with carboxylic group, ester group strongly stretched found at 1741.68 cm^{-1} . C-O group combined with carboxylic group, ester group strongly stretched at 1168.04 cm^{-1} (1260-1000 cm^{-1}). Alkanes (C-H) strongly stretched at 1459.71-1168.04 cm^{-1} .

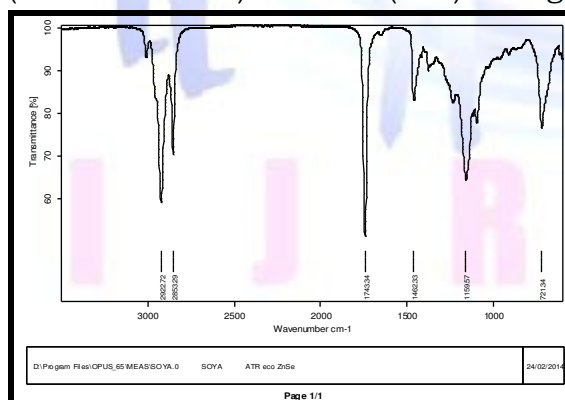


Fig.6 : FTIR spectrum of soyabean seed oil

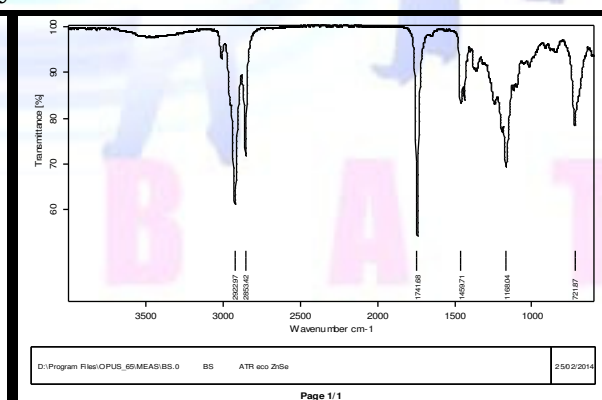


Fig.7: FTIR spectrum of biodiesel prepared from soyabean oil

Brown flax seed oil: Figure 8 shows the FTIR spectrum of brown flax seed oil. The spectrum was characterized with asymmetric and symmetric strong stretching vibrations of carboxyl group at 2853.21 cm^{-1} and aldehyde, ketones (C=O) group along with carboxyl group at 1742.94 cm^{-1} . C-O group combined with carboxylic group at 1160.21 cm^{-1} . C-H of alkanes are strongly stretched at 2922.63, 2853.21 and 1462.00 cm^{-1} . Figure 9 shows the FTIR spectrum of biodiesel using NaOH Catalyst. The spectrum was characterized with asymmetric and symmetric strong stretching

vibrations of carboxyl group in the range 3000-2500 cm^{-1} , along with the O-H stretching of the hydroxyl bonded with alcohol at 3600-3200 cm^{-1} (broad). Aldehyde, ketones (C=O) group along with carboxylic group, ester group strongly stretched at 1741.46 cm^{-1} . C-O group combined with carboxylic group, ester group strongly stretched at 1168.40 cm^{-1} (1260-1000 cm^{-1}). Alkanes (C-H) strongly stretched at 1458.27-1168.40 cm^{-1} .

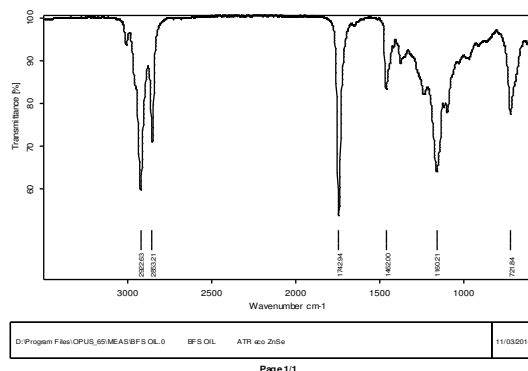


Fig.8: FTIR spectrum of brown flax seed oil

Conclusion:

In the present study, the extraction of oils from brown flax seeds, groundnuts, cotton seeds, cloves, soyabeans and coriander seeds were carried out by using soxhlet extractor. Different solvents for different seeds have been used such as water (Brown flax seed), ethanol (clove), petroleum ether (ground nut, coriander seeds), n-hexane (soyabean, cotton seeds). The extracted vegetable oils were used for the production of biodiesel. Characterization of oils as well as biodiesel was done using FTIR spectroscopy. Vegetable oils are a renewable and potential inexhaustible source of energy with an energetic content close to diesel fuel. In addition to being available locally, cheap and renewable, biodiesel can make a good substitute for diesel fuel. In addition the green aspect is that biodiesel is biodegradable and non-toxic. Byproduct i.e. glycerol can be used as an additive to make glycerin soap.

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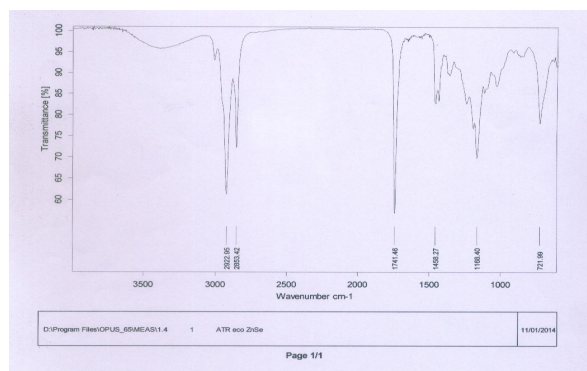


Fig.9: FTIR spectrum of biodiesel prepared from brown flax seed oil



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