Sodium Hydroxide is an Effective Catalyst for Synthesis of Bio-diesel from Swietenia Macrophylla Seeds

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Abstract—Increasing demand of conventional source of fuel makes people more conscious about fuel consumption. To make up the crisis of fuel, they start to search alternative source of fuel so that those source can substitute the present energy demand by some percentage. From this prospect in this study, we were carried out an experiment using some operating variables to determine the potential of Swietenia macrophylla for bio-oil production and the eventual making of liquid biodiesel products. The biodiesel was produced via trans-esterification process using methanol and catalyst as Sodium hydroxide. After characterization of the seed, extracted oil and produced biodiesel, the result shows that the seed has a moisture content of 12 mg/kg, about 47.55% by weight of seed was converted bio-oil, where biodiesel yield is 72% having a density of 0.885gm/cm^3 and the FT-IR analysis assure that the presence of alkanes, esters, aromatics, hydroxyl, and carbonyl in the biodiesel. Calorific analysis revealed that biodiesel has high heating value of 40.45 MJ/Kg. This investigation demonstrates that the seed from S. macrophylla are useable biomass in case of the production of biodiesel to meet the required energy demand.

Index Terms—Swietenia macrophylla, Extraction, Methanol, Trans-esterification, Sodium hydroxide, Bio-diesel, FT-IR.

1 INTRODUCTION

Due to the depletion of the world’s petroleum reserves and the increasing environmental concerns, there is a great demand for alternative sources of fossil fuel such as diesel and gasoline. Biodiesel is one kind of renewable fuel which has been in recent times used as best substitute of diesel fuel due to its useable properties in compression ignition engine without any modification [1]. Chemically, biodiesel is a mixture of methyl esters with long-chain fatty acids and is typically made from nontoxic, biological resources such as vegetable (seed) oils “[2], [6]”, animal fats “[7], [9]”, or even used cooking oils [10]. Seed oils are potential feedstock for biodiesel production and it can be environmentally produced in large extent [11]. Vegetable oils are comprised of edible and non-edible oils. In excess of 95% of biodiesel are manufactured from edible oils and properties of this formed biodiesel are considered to be used as substitute of diesel fuel [12]. However there is a composition between human consumption of edible oils and production of biodiesel from edible oils. The cause is that, many researchers emphasis their attention on the production of biodiesel from non-edible oils “[13], [15]”. S. macrophylla (mahogany) is one of the tropical trees that could potentially provide biofuel products. Mahogany seed oil is inedible [16]. The fatty acid ingredients of mahogany oils are: stearic acid (10.41%), palmitic acid (21.39%), oleic acid (64.62%) and unidentifiable acid (3.58%) [16]. Mahogany oil does not contain certain essential fatty acids; hence it does not have nutritional value. Particularly, more than 50% of the components of the seed of the S. macrophylla are carbon (48.14%) and hydrogen (6.4%), the elements composing the biofuel products [17].

At present time, there are several methods existing for the production of biodiesel fuel such as direct use and blending of raw oils “[18], [20]”, micro-emulsions process [21], thermal cracking “[22], [24]”, and transesterification [25]. Among these above methods, transesterification of seed or vegetable oils are most frequently used method. The key objective of this study is to investigate the biodiesel production rate from S. macrophylla seed oil and demonstration the variety of optimum operating conditions. The diverse factors which affecting the yield of biodiesel are also discussed.

2 MATERIALS AND METHODS

2.1 Collection of S. Macrophylla seeds

The S. Macrophylla seeds were collected from local area of Bangladesh. The seeds were taken from the capsules and sun dried for 8h, after that it was oven dried at 180°C for 5 minutes. A moisture analyzer (MS-70, A and D company Ltd.) was used for analysis of moisture content. The moisture content of the dried seeds is calculated using the following equation,

\[ Mc = \frac{W_1-W_2}{W_2} \times 100\% \]

Where, Mc is the moisture content, W1 is the weight of raw seed and W2 is the weight of dry seed.

2.2 Oil Extraction method

After blending, the dried seeds become grind form. The grind sample (mahogany seed) are prepared by blending is weighted. Three known procedures for extracting the oil from seed are (1) Expeller/press method, (2) Solvent extraction via
chemicals method and (3) Supercritical fluid extraction method [26]. The utmost common extraction method is the Soxlet extraction using hexane as a solvent. The extraction time range employed in this research was 3 to 6 h. [27] whereas the amount of hexane used was varied between 250 ml and 400 ml. The grid sample was taken in an extraction chamber. Then hexane (solvent) and sample with a ratio of 4:1 is poured into a round bottom flask. Soxlet apparatus is used for the extraction. At the end of this process, the extracted sample was separated. The solvent was evaporated and condensed to be recuperating in another funnel for reprocess in succeeding extractions.

The mass of the residual oil was measured and the percentage of oil in the original sample was calculated. The experiments were carried out at different extraction times and different volumes of hexane were employed. Since high proportion of oil is extracted, solvent extraction has become the utmost popular technique for abstraction of oils and fats [28]. The % of S. macrophylla oil extracted is calculated by using the equation below:

\[
\text{\% Oil extracted} = \frac{\text{Weight of extracted oil in grams}}{\text{Weight of sample in grams}} \times 100\%.
\]

2.3 Biodiesel production through Trans-esterification by hot plate

Biodiesel was produced by base catalyzed trans-esterification method via hot plate. The reaction was performed at 60°C through constant stirring for 1 hr. Fifty ml oil sample was placed in a two necked 250 ml round bottom flask equipped with a reflux condenser. The flask was transferred on an electric hot plate with a temperature controller and a thermometer. Sodium hydroxide pellets were used as catalyst, which dissolved with required amount of methanol (99.8% purity). Molar ratio of oil to methanol was taken from 1:2 to 1:8. Then 50 ml oil sample and catalyzed methoxide solution was replaced in the flask, after completing the reaction, the product was taken in an extraction funnel for about 12-15 hours. Two distinct layers were observed. Upper layer was methyl ester (biodiesel) and lower layer was combination of soap, crude glycerin, residual methanol and catalyst. After water washing and settling, the water and impurities were exhausted from the bottom of separating funnel and biodiesel was dried in a dryer at 100-105°C for about 30 minutes.

3 RESULTS AND DISCUSSION

3.1 Optimization of oil yield:

Time, temperature and solvent have a great influence on oil yield. By changing these parameters oil yield is also changed. To get maximum yield these parameters is needed to be optimized. The optimized conditions are discussed below.

3.1.1: Effect of hexane on oil yield

The hexane is widely used solvent in oil extraction, because of its idyllic functionality for maximum extraction [29]. It is non-polar and apparently dissolved non-polar compounds in the seeds following the chemistry rule that “like dissolves like.” It means nonpolar compounds in the seeds were dissolved by hexane and became part of the filtrate after the solid residues were separated. Figure 3 shows that the extreme yield is 47.55%, which is obtained at hexane to sample ratio 4:1 [30], whereas 35% yield was reported by Elkhaleefa and Shigidi at the same ratio. Since a maximum yield was found at a ratio of 4:1, this ratio was further used.

3.1.2: Effect of time on oil yield

Fig-4: Variation of oil yield (%) with time (hr). Condition: Sample 100 gm, Hexane to sample ratio 4:1, T=60°C.
Figure 4 shows percentage of oil yield with time at several amounts of hexane. In every case yield of oil increases through time and becomes maximum at 6 hr. This figure represents that increasing mixing time from 6 hr, the yield decreases. This result is similar to the findings of Mani et al. [31]. This phenomenon is due to low solvent density left in the sample after 6 hours [29]. However, the oil yield increased radically when the *s. macrophylla* seeds were heated from 1 hr until 6 hr because the diffusivities of the oil and solvent increases, which result in high oil yield. Therefore, the maximum yield could be achieved at a tinier retention time with an optimum temperature of 60°C.

### 3.2 Optimization of biodiesel production

Catalyst (NaOH and KOH), time and solvent have a great effect on biodiesel yield. By altering these parameters biodiesel yield is also altered. To get better yield these parameters is needed to be optimized. The optimized conditions are discussed below.

#### 3.2.1: Effect of amount of catalyst on biodiesel yield

Figure 5(a) shows the variation of yield (biodiesel conversion) with two different catalysts KOH and NaOH, where the catalyst was varied in the range of 0.1 g to 1.0 g. From figure it is clear that the biodiesel yield for NaOH catalyst is always higher than KOH catalyst, which is analogous to Cynthia et al [32]. The yield of biodiesel from 36.10% - 84.30% was obtained by Renato et al. [17].

Fig. 5(b) the yield of biodiesel ranges from 54% to 72% for NaOH and 52% to 69%, which satisfies the result of Renato et al. [17]. So, the optimum catalyst (NaOH) is 1%, which satisfies Atila et al [33]. Here it is obvious that biodiesel yield increases with the increase in amount of catalyst (from 0.3g to 0.8g). This result is analogous with the findings of Kumar et al. [32] where they showed that biodiesel conversion decreases due to soap formation [33].

#### 3.2.2: Effect of biodiesel and yield with variable Time

Figure 6 give a concept of biodiesel production as well as yield percentage with time. It shows that the yield increases with the increase in time and gets area of stability after definite time. As it is shown that at 60 min the yield is 55.7% while at 90 min it increases to 70.3. This happens because the system gets enough time for a complete reaction to occur. From this figure an optimum time 90 min was obtained.

#### 3.2.3: Effect of methanol on bio-diesel yield

Figure: 7 shows that the bio-diesel yield increases with raw oil to methanol ratio and goes to maximum at 1:6 ratios then it decreases. Alcohol to oil ratio is one of the most significant factors affecting yield of biodiesel as well as biodiesel production cost [34]. The stoichiometric ratio of oil to alcohol for the transesterification is 1:3 and the reaction is reversible, higher molar ratios are required to increase the miscibility and to enhance the contact between the alcohol molecule and the triglyceride. In practice, to shift the reaction toward completion, the molar ratio should be higher than that of the stoichiometric ratio [35]. Further to break the glycerin–fatty acid linkages during transesterification of triglycerides to biodiesel, excess methanol is required [36]. Therefore, higher alcohols to oil molar ratios give rise to greater alkyl ester conversion in a shorter time [37]. Moreover, increase in the amount of alcohol to oil increases biodiesel yield and biodiesel purity.
3.3.1: Properties of Biodiesel:

![Table 1: Properties of Biodiesel Obtained from S. macrophylla Seeds Oil.](image)

Table 1 compares some basic properties of the synthesized mahogany biodiesel and the ASTM D6751, 482, 7467 standard for biodiesel fuel (B100) \[38\], [40]. The major important property of biodiesel is viscosity which is the measure of biodiesel’s ability to flow. Thickening of lubricating oil is the consequence of high viscosity and due to this reason this property bounds the direct application of vegetable oils in diesel engine. As apparent in Table 1, the kinematic viscosities of mahogany biodiesel and ASTM standard biodiesel are very close, and fall within the range specified the ASTM D6751 standard. Fuel density directly affects fuel performance, as some of the engine properties; the density of the fuel also affects the quality of atomization and combustion. The densities of mahogany biodiesel are high and also range within ASTM standard. Subsisting moisture in biodiesel progress the free fatty acid concentration which can be the cause of corrosion of metallic parts of the fuel engine system. Minimum moisture level is suitable for biodiesel. Moisture content of S. macrophylla is 0.12mg/kg where ASTM standard range is 0-5000mg/kg. A quantity of ash is formed consisting of unburned hydrocarbons and inorganic impurities. Minimum ash content is also suitable for biodiesel. Ash content of S. macrophylla is 0.003%. The flash point does not affect the combustion directly; higher values make fuels safer with regard to storage, fuel handling and transportation. For biodiesel the range of flash point is 100-170°C where our S. macrophylla biodiesel’s flash point is 156°C. Pour point overestimates minimum operating temperature at which the fuel can be used. The pour point values depend on the feedstock the biodiesel was produced from. Pour point values are between -15 °C to 10 °C and S.macrophylla biodiesel’s pour point is 7 °C. The cloud point is the most commonly used measure of low-temperature operability of the fuel. The biodiesel cloud point is typically higher than the cloud point of conventional diesel. The cloud point of biodiesel also depends on the nature of the feedstock it was obtained from -3-12°C and our S.macrophylla biodiesel’s cloud point is 8°C. The acid value (AV), also called neutralization number. In biodiesel sample, the acid value is determined to enumerate the existence of acid moieties. From ASTM standard the maximum acid value of biodiesel is 0.80 where S. macrophylla biodiesel’s acid value is 0.54. A too high total of free glycerin can cause functioning problems at abridged temperatures and fuel filter clogging. This parameter can also be used to measure the cleanliness of the biodiesel. The volume of S. macrophylla biodiesel’s free glycerol is 0.005% which is lower the maximum ASTM standard.

3.4: FTIR analysis

![Figure 8: Fourier Transform Infrared Radiation (FT-IR) Spectrum of biodiesel from S.macrophyllaseed extract.](image)

3.4.1: Result of FT-IR

The functional groups present in the biodiesel extracted from the S. macrophylla seeds were identified via FT-IR analysis. The figure 8 Fourier Transform Infrared Radiation (FT-IR) spectrum of biodiesel from S.macrophylla seed extract. A broad absorption band observed at 3008cm⁻¹ is attributed to the O-H stretching of hydroxyl group from alcohols. The observed peak at 2924 cm⁻¹ is caused by C-H stretch suggesting the presence of alkanes. The C=O stretching at 1745cm⁻¹ and 1163 cm⁻¹ indicate presence of ether and carbonyl compound while another bond at 962 cm⁻¹ indicate bending aromatic compound. The peak at 1030 cm⁻¹ indicate the possible presence of sulphoxide at stretching Sulphinyl (S=O) compound.

4 CONCLUSIONS

This work explored the potential of using the seeds of S. macrophylla in the production of biodiesel. The physicochemical characteristics of the seed oils from S. macrophylla are helpful to identify the quality of oil and biodiesel products for possible industrial or commercial uses. The refining of crude mahogany oil leads to the reduction of specific gravity, moisture content, viscosity, and acid value. Decreasing of these properties can be certified to the taking away of phospholipids via degumming with phosphoric acid carried out by neutralization with potassium hydroxide solution, and adsorption of additional contaminant by activated charcoal. The S. macrophylla seeds, after sun drying, contain acceptable mois-
ture content ready for oil extraction. The oil content of the S. macrophylla seed ranges from 38.3% to 47.05% by weight. The optimal condition for better yield for oil operating time 6 hr. sample to hexane ratio is 1:4, and for bio-diesel Sodium hy-

REFERENCES

[39] European Commission, 2007; NREL, 2009; Prankl, et al., 2004