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Technical Complications in Expelling and Converting Domba Oil into Biodiesel

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Abstract- **Desiccated resinous 'Domba' (*Calophyllum inophyllum* L.) kernels were mechanically expelled to obtain oil. The highly viscous cold pressed oil was then converted into biodiesel using four different transesterification protocols in a newly developed programmable biodiesel reactor. This paper describes the difficulties associated with expelling and converting Domba oil from the kernels, and the efforts made to overcome the problems. Amongst the four methods tested, 4-stage transesterification protocol was found to yield better quality biodiesel than the other protocols.**

I. INTODUCTION

Calophyllum inophyllum L. (Clusiaceae) commonly known as 'Alexandrian Laurel', 'Beauty Leaf' or 'Domba' (in Sri Lanka) is essentially a littoral tree of the tropics, occurring above the high-tide mark along sea coasts of northern Australia, and extending throughout Southeast Asia and southern India [1]. 'Domba' Tree is a medium to large evergreen tree that averages 8–20 m in height with a broad spreading crown of irregular branches. The tree supports a dense canopy of glossy, elliptical leaves, fragrant white flowers, and large round nuts (Fig.1) [2].

Figure 1: Fruits and Oil bearing kernels of *Calophyllum inophyllum* L

The tree has many favourable features to be used as a source of biodiesel. It fruits excessively [2], the kernel which is non-edible contains 55-70% oil [3], and the oil has been traditionally used as a lamp oil [4]. Seed oil of *C. inophyllum* is dominated by unsaturated fatty acids [5], [6]. Fatty Acid Methyl Esters (FAME-biodiesel) of *Calophyllum inophyllum* also conforms to US, European and German biodiesel standards [7], [8] and [9]. The kernels also contain <20% resin [3] and as a result it is often difficult to expel oil using conventional techniques.

Cold pressed oil is favoured in biodiesel production, as non-polar solvent (e.g. n-hexane) extraction can contain other non-polar compounds that can interfere with transesterification reactions. Cold press extraction of resinous kernels has rarely been reported in scientific literature.

Chemical conversion of vegetable oils into fatty acid methyl esters is known as transesterification [10], [11]. Conversion of highly acidic and viscous 'Domba' oil into clear light biodiesel is not easily accomplishable with conventional transesterification protocols [8]. A new programmable biodiesel reactor was therefore designed, constructed and tested using different transesterification protocols.

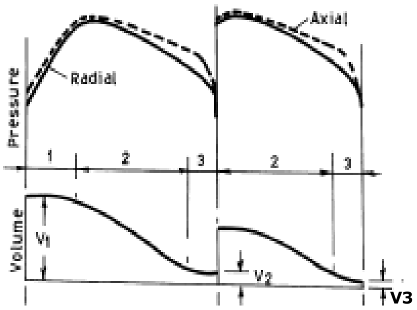
II. MATERIALS AND METHODS

A. *Seed collection*

Seed collection was carried out in Sri Lanka between June and August 2008 from following locations; Matara (5° 56' 55N, 80° 32' 34E), Colombo (6˚ 54'N, 79˚ 50' E), Anuradhapura (08˚ 20' 60" N, 80˚ 22' 60") and Kurunegala (7°45′N, 80°15′E). Fruits were deshelled and kernels were dried at 40 ºC for 18 days.

B. Expeller design

Cold expression of oil can be achieved using a screw press. However for efficient extraction the screw press should generate adequate pressure (4-35 MPa) to compress kernels and rupture cell walls to extrude oil through the slits provided along the barrel length (Fig. 2) [12] and (Fig.3).



1- Feed section
2- Ram section
3 -Plug section
V1 Feed volume
V2-Volume of cake after primary compression
V3-Volume of cake after secondary compression

Feed point — Discharge at primary section endpoint — Discharge at secondary section endpoint

Fig. 2. Pressure and volume variation based on the theory of Ward [12].

Considering the above requirements, a screw press was deigned and developed (Fig. 3) at Uva Wellassa University, Sri Lanka using the following configuration.

Drive train	Motor-> Belt drive->Gear box-> Screw

TABLE 1: Specifications of the developed expeller

Parameter	Dimensions
Motor	5.6 kW/3 Ph/ 1440 rpm
Belt drive ratio	1:4
Gear box ratio	1:3
Total ratio	1:12
Outer diameter of the barrel	85 mm
Inner diameter of the barrel	52 mm
Shaft length	250 mm
Outer diameter of the shaft	50 mm
Depth of the cut	7 mm
Helix angle	60^o
Pitch	25 mm
End taper	10%

The tapering end of the screw was expected to provide adjustable compression ratios by changing clearance. This was achieved by moving the shaft back and forth.

$$\text{Compression Ratio (R)} = \frac{D_B - D_F}{D_B - D_E}$$

Where, D_B; diameter of the barrel, D_F; root diameter of the shaft at feeding end, D_E: root diameter of the shaft at start of the plug section.

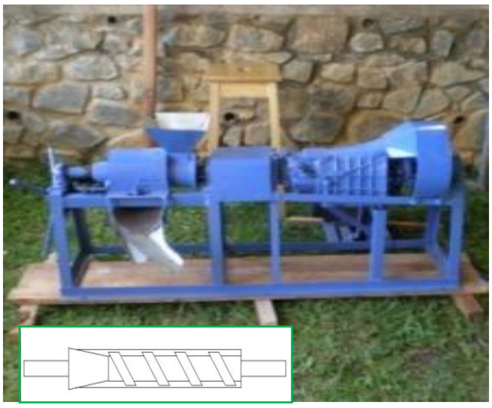


Figure 3: The developed oil expeller and the screw shaft

C. Oil extraction

Prior to expressing oil from *Calophyllum* kernels, the oil press compression ratio was optimised by using 2 kg of dried coconut and 2 kg of *Calophyllum* kernels. The dried kernels (moisture content 8%) were then fed to the expeller at constant rate (3 kg/ hr) while monitoring the temperature of the screw press. The oil was collected separately at every 15 mins wile recording the temperature. The cake was fed in two cycles to get the maximum oil output.

D. Reactor design

An automated biodiesel reactor was designed, developed at the Uva Wellassa University, Sri Lanka. The reactor (Fig. 4) consisted of three main chambers made up of high dense polyethylene (HDPE). They were namely; reaction chamber (A), a mixing chamber (B) and a settling chamber (C). In reaction chamber heat was generated by a 2000 W heat element, and the temperature was controlled by a J type thermo couple. Other components included a methanol condenser (D), small AC motor (0.47 W /1 Ph/ 2831 rpm), two pumps, control panel with LCD display and a microcontroller PIC16F877. Microcontroller was used to regulate the temperature, stirring speed and the reaction time.

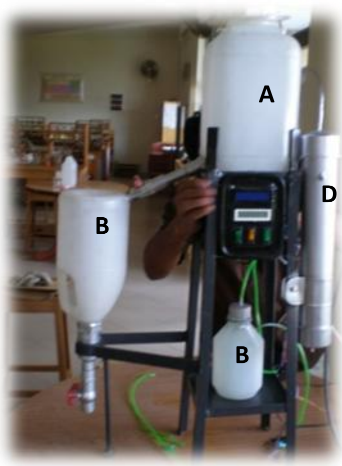


Figure 4: The automated biodiesel reactor

E. Conversion

Four different transesterification protocols (viz. base catalysed, acid catalysed, 3-stage and new 4-stage) were carried out using the designed biodiesel reactor (Fig 4), and the resulting FAME were tested for physicochemical properties (with 3 replications) using ASTM methods; D 2500 for cloud point, D 97 for pour point and D1298 for density. After testing for normality and homogeneity of error variance the data were subjected to ANOVA by using GENSTAT edition 11.1.

III. RESULTS AND DISCUSSION

A. Oil extraction

At maximum compression ratio (9:1), creamy extrusion was observed to flow between the slits of the barrel. At minimum compression ratio (2.8:1) under-crushed kernels were collected with the cake. Best compression ratio was found to be 7.4: 1. At this ratio, the viscous dark greenish oil also contained a considerable amount of residue. Adjustable 1mm wired net cover was fixed to the barrel in order to reduce the creamy residue mixing with the oil.

The ideal operating temperature was found to be 65 ^{o}C (Fig. 5).

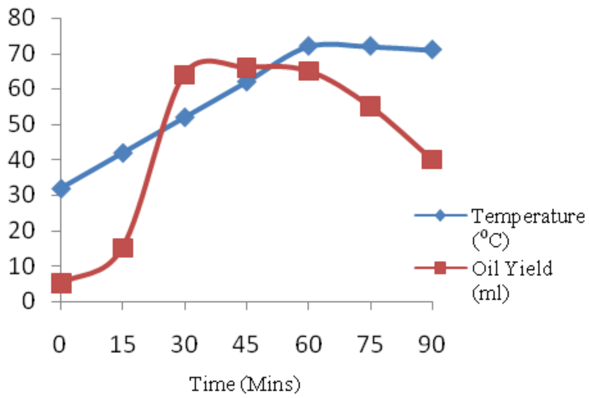


Fig. 5. Oil yield at different temperatures

The barrel temperature of the expeller increased with the addition of kernels and remained constant between 65 ^{o}C-72 ^{o}C. Maximum oil yield was obtained at 65 ^{o}C. Oil content declined after 75 mins due to the pressure reduction in the barrel caused by the decease of load in ram section. Later the oil yield increased gradually after gaining the optimum load.

Fig. 6. Hydraulic press

The oil was then filtered by using a cheese cloth and the residue was fed to a hydraulic press (Fig. 6) to increase oil yield.

B. Oil to biodiesel conversion

Figure 7 shows that the colour and the clarity of the biodiesel vary with the method used. The Method 4 (4-stage transesterification protocol) yielded the clearest biodiesel.

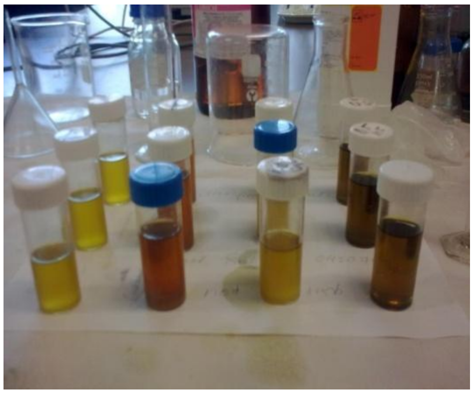


Fig. 7. FAME derived from different methods (from left; base catalysed, acid catalysed, 3-stage, 4-stage).

Transparency of biodiesel decreased in following order method 4> method 3> method 2 > method 1. The quantities of different reactants and reaction conditions used in the four methods, and the properties of the biodiesel obtained from the above methods are shown in Table 2.

As shown in Table 2, FAME derived from different protocols differed significantly ($P<0.05$) in pH, conversion %, cloud point, pour point and the amount of by-product. However densities of resulted FAME were not found to be statistically significant.

Conversion rate for Method 3 and Method 4 were significantly higher than other methods. Could point, pour point and pH of the FAME derived from the method 4 were found to correspond more with the ASTM biodiesel standards than the resulted FAME of other methods.

TABLE 2. Comparison of four transesterification protocols; Method 1;- base catalysed, Method 2;- acid catalysed, Method 3 – as per Sahoo et al. [8] and Method 4;- the new method.

Parameter	Meth1	Meth2	Meth3	Meth4
Calophyllum oil (ml)	100	100	100	100
Methanol (ml)	25	25	35	35
Ortho phosphoric acid (ml)	0	0	0.5	0
Toluene (ml)	0	0	5	5
Sulphuric acid (ml)	0	0.65	0.65	0.65
Propanone (ml)	0	0	0	0.5
Potassium hydroxide (g)	0.65	0	0.9	1.25
Reaction temperature (°C)	60	60	66	64
Stirring speed (rpm)	550	500	500	550
Reaction time (min)	180	45	240	240
Settling time (hr)	15	8	17	18
Total Product (ml)	111.9a	110.5b	129c	126.3d
FAME yield (ml)	77a	83b	86.25c	88.2c
By product +(ml)	34.3a	27.3b	42.6c	38.3d
Conversion (%)	77a	83b	86c	88c
pH of FAME	7.6a	5.9c	6.9b	7.1b
Density (kg m^{-3})	844	869	842	827
Cloud Point (°C)	11.4a	10.5b	10.8c	9.8d
Pour Point (°C)	6.2a	4.5b	4.3b	3.9c

*within a row means sharing the same letter do not differ significantly (P<0.05), +; also includes impurities

IV. CONCLUSIONS

Extraction of 'Domba' oil is difficult to be accomplished with a conventional screw press. Modifications that were made to the expeller and the extraction process resulted in appreciable increase in oil yield. Continuous extraction can be achieved by using medium compression ratios (7.4:1) and ensuring that the operating temperature does not exceed 60 ºC. A newly developed programmable biodiesel reactor was found to be satisfactory to convert Domba oil to biodiesel, and the new 4-stage transesterification protocol produced the most suitable quality biodiesel.

ACKNOWLEDGMENTS

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