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DIMENSIONAL PROPERTIES AND PYCNANTHUS SEED OIL EXTRACTION VIA RESPONSE SURFACE METHODOLOGY: A NOVEL POTENTIAL BIOFUEL CANDIDATE

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Abstract: The need to effectively design and fabricate in-situ batch reactor and deshelling equipment as well as seek novel inedible feedstock which will not stress food crop has necessitated the assessment of dimensional properties of pycnanthus seeds and conduct physical properties of oil extract. Pycnanthus (Pycnanthus Angolensis) seeds, as inedible seed oil in food industry, remain underutilized. The oil as a novel oil has not been assessed for biofuel application via determination of physical properties of the seeds and oil. Dimension/engineering properties of the pycnanthus seed (PCS) were determined. The effect of extraction process such as hexane to seed ratio between 2 to 6 ml/g and extraction time from 1 to 8 h were investigated on the yield of pycnanthus seed oil (PSO) using Response Surface Methodology (RSM). Basic fuel related properties at the optimum condition were obtained following standard laboratory procedures. The dimensional properties of the PCS indicated that the seeds are ellipsoids. Optimization predicted PSO yield of 38.64% at the optimal level of 6.0 ml/g of hexane to seed ratio and 8 h for extraction duration. Fuel properties of the PSO were almost within the specification of the American Oil Chemists' Society and the highly saturated value of the fatty acid composition (FAC) of the PSO indicate that the biodiesel from the oil will exhibit better oxidation stability and reduced NOx emission. The bio-data from the engineering properties of the PCS can be further explored to fabricate oil screw expeller, des-shelling equipment, in-situ batch reactor and other farm machinery equipment. The GC-MS analysis of the FAC of the PSO indicates that the oil can be employed for biodiesel production for a reduction in exhaustion emission and obtain stable oxidation stability. The extraction parameters can be scaled up industrial to extra oil from the novel seed for necessary biofuel applications.
Keywords: Response Surface Methodology; optimization; pycnanthu seed oil; properties; dimension; yield

1. INTRODUCTION

The demand for edible lipid feedstock for biofuel production is on the increase and this has greatly stressed food production supply [1]. The scarcity has been attributed to a diversification of food crops to biodiesel production [2]. As a result, researchers have been motivated to search for lipid feedstocks which are non-edible so as to curtail competition in vegetable oil industries for biodiesel sustainability. Non-edible oil has emerged as a feasible feedstock for biodiesel production as it will not stress food crop supply [3]. Several researchers [4-6] have linked reductions in the price of biodiesel production to the utilization of inedible seed oils for bio-lubricant production. However, as biodiesel is gaining global attention, information regarding the physical/dimensional properties of the seeds has been remarked to be relevant for proper designing of expeller and equipment processing [7]. In addition, Chukwu and Sunmonu [8] emphasized that engineering properties are useful for effective processing handling machinery part, storage, structure and processing harvest. Researchers [9-12] have reported that dimension properties of seeds are relevant for design of machinery equipment. They further remarked that machinery elemental units such as hopper, press auger, and press barrel can be properly designed if dimensional properties are incorporated. Owing to the relevance of the dimensional properties of the seeds for wide varieties of design purposes, researchers have documented their findings. Roseller seeds [13], Kohanz apple fruit [14] palm nut [15], rubber seed [17], indigeneous seed [18], etc were reported. To the best of our knowledge, information related to the engineering properties of pycnanthus seed and extraction optimization has not been reported.

The Pycnanthustree growth is up to 15 to 25 m tall and mostly found in West and Central Africa [19]. Pycnanthusangolensisbelongs to the myristicaceae family. It is often referred to as African nutmeg and wild nutmeg. The extract of the plant are reported to have several medical applications [20]. The juices from the leaf are used as mouth wash to treat oral thrush inchildren while the seed fat is suitable for treating fungalskin infections and the extracted of the seeds has been reported [21] to be suitable for soap and candle production. Aside dimensional properties, physicochemical analysis of oil prior biodiesel/bio-lubricant production have been hinted [22] to be relevant for an effective production. The physicochemical properties such as density, viscosity, saponification value, acid value, e.t.c have been documented for several non edible seeds such as jatropha seed oil [23], rubber seed oil [24], tobacco seed oil [25] and castor seed oil [26]. Moreover, optimum extraction conditions have been highlighted for several seed oils such as sage oil [27], hemp seed oil [28], Gmelina seed oil [29], Bauhinia monandra seed oil [30]. In view of the usefulness of the pycnanthus seed and its extracts for biochemical and biofuel applications, information related to proper design of machinery equipment is quite relevant. In the present study, the influence of the process variables such as extraction time and seed to hexane ratio were optimized by Design of Experiment (DOE) using Response Surface Methodology (RSM). Mathematical corrections for extraction of oil were formulated to describe the impacts and relationship between the extraction parameter. Characterizations of collected oil samples were analyzed to assess its suitability for bio lubricant product.

2. EXPERIMENTAL SECTION

The pycnanthus seeds were obtained from the experimental field of the Okitipupa farm land, Ondo state, Nigeria. Prior commencing the extraction process, the seeds were cleaned and passed through a Tyler sieve in order to remove foreign material. Vernier caliper was used to measure axial dimensions of the seeds while soxhlet extraction unit was set-up to extract oil from the Pycnanthus seeds. Fuel related properties such as density, viscosity, flash point moisture content and specific gravity were determined in accordance with ASTM standard protocols.

The three axial dimensions of the seeds namely: length (x), width (y) and breadth (z) were measured using a vernier caliper (See Figure 1). Ten random samples of the Pycnanthus seeds were measured and the average dimensions repeated. The unit mass, m (g) was computed using an electronic scale (Model Adam capacity 150 g, accuracy and 0.001g). Ten random samples from each seed bulk were taken in five replicates and measured. The unit mass of the seed was calculated using equation (1).

Figure 1. (a) Pycnanthu seed; (b) Measurement of pycnanthus seeds' dimensions

$$Da = \frac{m_{AV}}{10} \quad (1)$$

where m and m_{AV} are the respective unit mass (g) and average mass (g).

The arithmetic mean diameter, Da and geometric diameter, Dy of the seeds were calculated from the geometrical dimensions as repeated elsewhere [31-33] using equations (2) and (3), respectively.

$$Da = \frac{(x+y+z)}{3} \quad (2)$$

$$Dy = (xyz)^{1/3} \quad (3)$$

The sphericity of the seeds, ϕ, was calculated based on the isoperimetric property of a sphere as reported elsewhere [33] and computed using the expression highlighted in equation (4).

$$\phi = \frac{(xyz)^{1/3}}{x} = \frac{Dy}{x} \quad (4)$$

The unit volume of the pynanthus seeds, Vu (cm^3) was computed using equation (5)

$$Vu = \frac{4\pi}{3}\frac{abc}{1000} \quad (5)$$

The bulk density of the seeds were determined by weighing the seeds and filling a $100\ cm^3$ capacity measuring cylinder and the bulk density was calculated using equation (6).

$$p_b = \frac{m}{v} \quad (6)$$

where P_b, m and v are the bulk density (g/cm^3) mass (g) of the seeds and volume (cm^2) of the seed, respectively.

The solid density, p_b (g/cm^3) was determined following the procedure stipulated elsewhere [33] and evaluated using equation (7)

$$p_s = \frac{m}{nv_u} \quad (7)$$

where p_s is the solid density (g/cm^3) and n is the number of seeds

The porosity, $\mathcal{E}$ (%) of the seeds was evaluated as outlined elsewhere [33] and evaluated in equation (8)

$$\mathcal{E} = \left(1 - \frac{p_b}{p_s}\right) 100 \quad (8)$$

The approximate surface area of the seeds, $s\ (mm^2)$ was determined using the approximate formular of a general tri-axial ellipsoid as outlined in Refs. [34-35] as indicated in equation (9)

$$S = {}^{4\pi p}\sqrt{\frac{a^p b^p + a^p c^p + b^p c^p}{3}} \quad (9)$$

where p =1.6075

where specific surface area of the seeds s_s(cm²/cm³) was calculated using equation (10) in accordance with the reports of Ref. [33].

$$s_s = \frac{Sp_b}{m} \quad (10)$$

where m is the mass (g) of one unit of seed

Extraction Optimization

The extraction of oil from the pycnanthus seed was conducted using a soxhlet extraction set-up depicted in Figure 2 and the procedures outlined by Waheed *et al.* [36] was adopted. The effect of pycnanthus hexane to seed (x_1) and the extraction time (x_2) was investigated using Response surface Methodology (RSM) and the detail of RSM was outlined elsewhere [37]. The range and levels of the parameter studied are highlighted in Table 1.

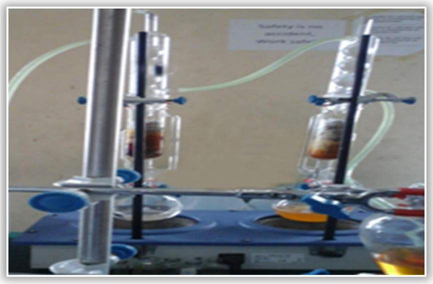

Figure 2. Soxhlet unit for pycnanthus seed oil extraction

Optimization of the extraction process was conducted with the RSM. The regression and graphical assessment of the data were investigated by using design expert. The maximum value of the oil yield was selected as the response of the design experiment. To assess the analysis of the variable, statistical analysis of the model was checked. The oil extracted was analysis using standard laboratory protocol. The GC-MS analysis of the oil was also conducted.

Table 1.Experimental range and levels for the extraction variables

Variables	Symbols	Range		
		-1	0	+1
Hexane to seed ratio	x1	2	4	6
Extraction time (hours)	x2	1.0	4.5	8.0

Physical Properties of Seeds

Basic geometric properties of pycnanthus seeds are presented in Tables 2. The unit mass of pycnanthus seed, (PCS) (0.88 g) was higher than that of palm kernel seed (PKS) (0.87 g) but lower than jatropha seed (JS) (0.78 g). Oil of higher unit mass has been reported to possess higher oil yield [38]. Therefore, higher oil quantity can be crushed from PCS.

Table 2. Basic geometric properties of pycnanthus seed

Seeds type	Unit mass m (g)	Length, x (mm)	Width, y (mm)	Breadth, z (mm)	Arithmetic diameter Da (mm)	Geometric diameter Dg (mm)	Unit volume Vu (cm^3)
[a]Pycnanthus seed	0.88±0.105	20.56±0.907	11.81±0.725	11.81±0.725	14.73	14.21	1.50
[b]Palm kernel seed	0.87	18.98	11.18	9.81	13.11	12.49	1.02
[c]Jatropha seed type (iv)	0.78±0.050	18.45±0.71	11.29±0.37	9.04±0.45	12.91±0.24	12.32±0.31	0.98±0.31

The length, width and breadth of PCS (20.56 mm, 11.81 mm, and 11.81 mm, respectively) are very close to those of PKS (18.98 mm, 11.18 mm, and 9.81 mm) and JS (18.45 mm, 11.29 mm and 9.04 mm, respectively). Owing to the close nature of the basic geometric characteristics of the PCS to those of the seeds mentioned there in, designing of processing equipment such as cleaning, sorting, packaging and storage can be attained.

The artithmetic diameter and geometric diameter of the PCS (14.73 mm and 14.21 mm, respectively) are higher than those of PKS (13.11 mmand 12.49 mm, respectively) and JS (12.91 mm and 12.32 mm, respectively). The design and fabrication of farm machinery may be influenced by the arithmetic and geometric diameters. The unit volume of the PCS (1.50cm^3) is higher than those of PKS (1.02 cm^3) and JS (0.98 cm^3).

Complex geometric properties of pycnanthus seed and its comparison with those of jatropha seed (JS) and palm kernel seed (PCS) are depicted in Figures 3 to 8. As oberved in Figure 2, the sphericity of the PCS (69.11%) is higher than those of JS (66%) and PKS (65.81%). The sphericity of the PCS (69.11%) is very close to those of PKS (65.81%) and JS (66.0%). PCS cannot be fully described as spherical seed based on the remark highlighted by Gamaya*et al.* [39].

The bulk density and solid density of PCS (0.36 g/cm^3 and 0.58 g/cm^3, respectively) are much lower than those of the PKS (0.61 g/cm^3 and 0.85 g/cm^3, respectively) and JS (0.44 g/cm^3 and 0.79 g/cm^3, respectively), as presented in Figures 3 and 4, respectively. Due to the lower value of the bulk density and solid density associated with the PCS, design of press hopper has to be properly effective in order to achieve effective oil extraction from the screw press [40-41].

The porosity of PCS (38%) is lower than that of JS (43.3%) but higher than PKS (28%) (See Figure 6). This implies that amount of pores in the bulk material is higher than PKS but less than that of JS. Air circulation might be reasonable in PCS compared to PKS and JS.

It was oberved that the surface area of PCS (666.55 mm^2) in Figure 6 exceed those of JS (53.2 mm^2) and PKS (524.55 mm^2).

The specific surface area of PCS (2.73$\mathrm{cm^2/cm^3}$) is lower than those of PKS (3.68 $\mathrm{cm^2/cm^3}$) and JS (3.0 $\mathrm{cm^2/cm^3}$), as drawn in Figure 8. This might affect the design of sieves for grading of PCS.

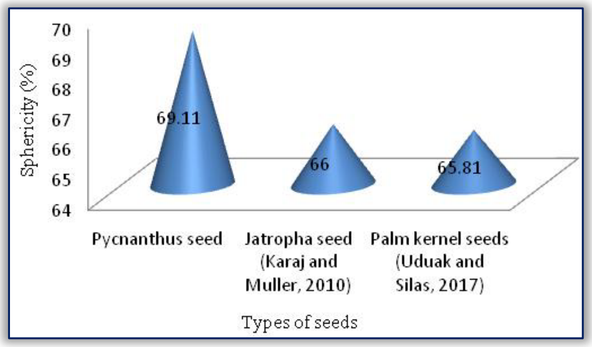

Figure 3. Spericity of pycnanthus seed with other oilseed

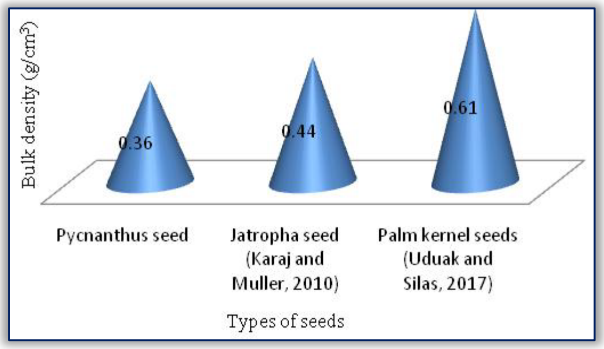

Figure 4. Bulk density of pycnanthus seed with other oilseed

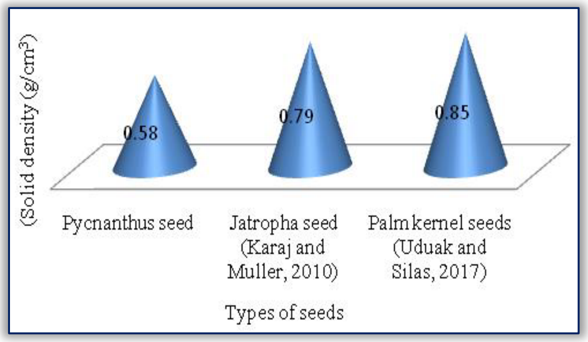

Figure 5. Solid density of pycnanthus seed with other oilseed

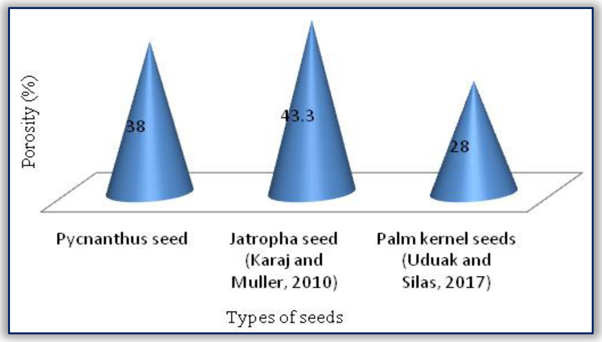

Figure 6. Porosity of pycnanthus seed with other oilseed

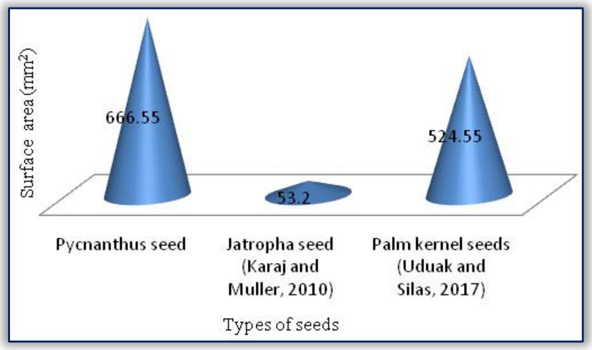

Figure 7. Porosity of pycnanthus seed with other oilseed

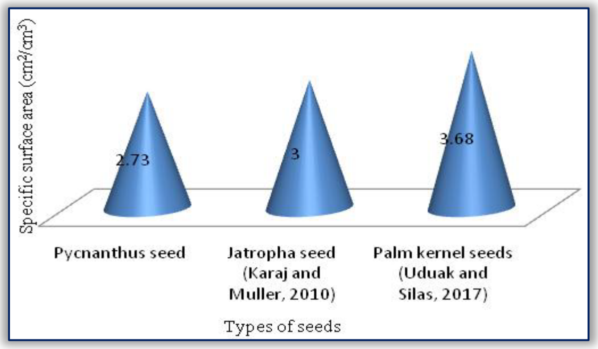

Figure 8. Porosity of pycnanthus seed with other oilseed

3. EXTRACTION OPTIMIZATION BY RESPONSE SURFACE METHODOLOGY

Analysis of Variance (ANOVA) for Model Fitting

Response surface Methodology was adopted in order to optimize the influence of extraction variables on oil yield. The experimental ranges for the independent parameters are highlighted in the previous section. The produced parameter and experimental generated data are presented in Table 3.

As can be noticed from Table 3, the significant for the oil yield varies. The coefficient of variation (% C_V = 3.79) implies that the results of the quadratic model were reliable. The obtained value (R^2 = 0.8861) indicates that 88.69 of the experimental data concurs with the predicted expressed in equation (12) and the actual represented by equation (13).

Table 3. Experimental design and produced yield

	Code		Response		
	X_1	X_2			
Run	Ratio (hexane/seed) (g/ml)	Time (h)	Actual yield (%)	Predicted yield (%)	Residue
1	0	0	32.00	32.48	-0.48
2	0	-1	37.30	38.30	-1.00
3	0	0	39.00	37.73	1.27
4	1	-1	39.40	38.65	0.75
5	1	1	33.40	34.19	-0.79
6	-1	0	37.80	37.56	0.24
7	-1	-1	33.10	31.62	1.48
8	-1	1	32.40	34.42	-2.02
9	0	0	31.40	32.11	-0.71
10	0	0	32,40	32.11	-0.11
11	0	1	33.00	32.11	0.89
12	1	0	32.40	32.11	0.29
13	0	0	32.30	32.11	0.19

Predicted expressed in equation (12) and the actual represented by equation (13).

$$\text{Yield }(\%) = 32.11 \pm 1.88A + 1.40B + 3.76A^2 + 0.94095R^2 - 1.22AB \quad (12)$$

$$\text{Yield }(\%) = 40.359 - 5.898R + 0.4286t + 0.94095R^2 + 0.07460t^2 - 0.1750R \quad (13)$$

where A (time (h) and B (hexane to seed ratio (ml/g) in design of experiment. In the same vein, t (time (h)) and R (hexane/seed (ml/g) are actual variables in the model to predict the oil yield.

The variation between the studied process parameters and the oil yield are depicted in Figure 9 (3D-plot and perturbation). The oil yield increased with the increase in the extraction time beyond 4.50 hours extraction time and the yield was observed to increase faster when the ratio of the hexane to seed is beyond 4.00 ml/g. The increase in the oil yield with the extraction time has been attributed to effective interaction between the solvent and solute [24].

Table 5: Analysis of variance for the quadratic model.

Source	Sum of squares	Degree of freedom	Mean square	F. value	P. value
Model	91.70	5	18.34	16.89	0.0034
A-time	17.00	1	17.00	10.09	0.0156
B-ratio	11.76	1	11.76	6.98	0.0333
AB	6.00	1	6.00	3.56	0.1010
A^2	39.13	1	39.13	23.23	0.0019
B^2	2.31	1	2.31	1.37	0.2803
Residue	11.79	7	1.68		
Lack of fit	10.42	3	3.47		
Pure error	1.37	4	0.34		

Corrected total standard deviation =1.30; Mean =34.37 Coefficient variation (%) = 3.79; R^2=0.8801 AdjR^2=0.8047

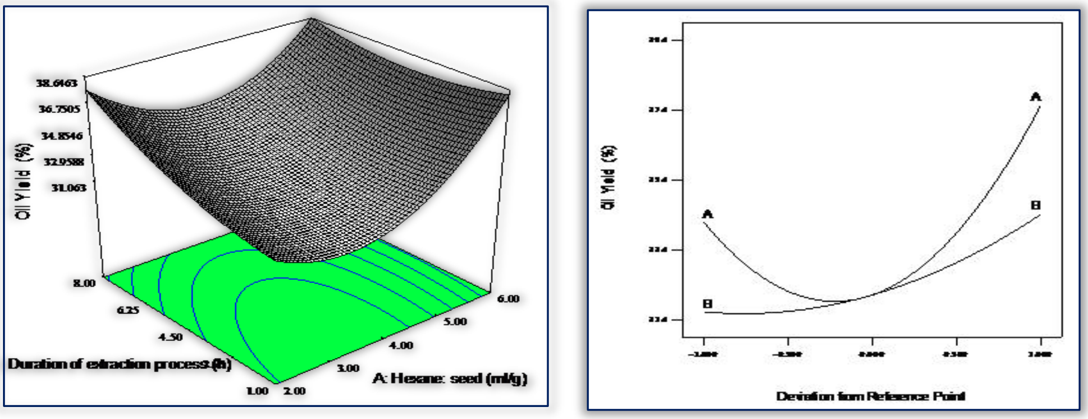

Figure 9. Variation of oil yield and extraction parameters, extraction duration and solute to hexane ratio; (b) variation from reference positions

The optimal values selected from the regression model expressed in equation (12) for selected parameters were determined by numerical method. The model produced optimal oil yield (38.64%) at 8 hours extraction time and 6 ml/g of hexane to seed with desirability of 0.91 (Figure 10).

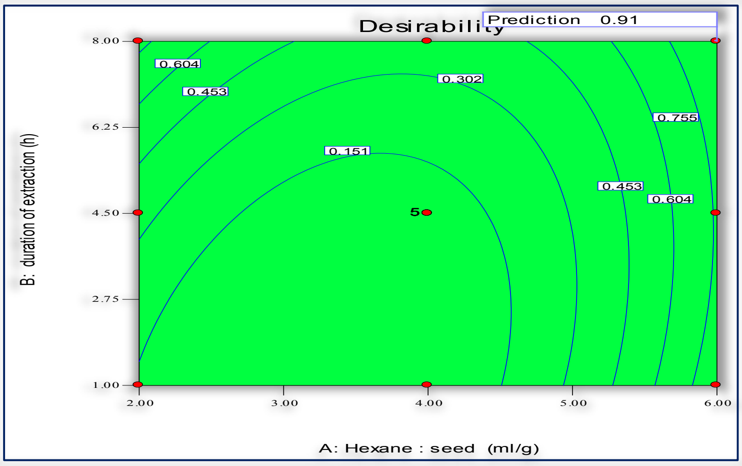

Figure 10. Variation of time of extraction versus ratio of hexane to seed with desirability

Characterization of Pycnanthus Seed Oil

The assessment of pycnanthus seed oil (PSO) was conducted by determining the physical and chemical properties, as presented in Table 6. The PSO had density and flash point of 978.0 kg/mm^2 and 220 °C, respectively. The acid value and %FFA of the PSO were 4.77 mg KOH/g and 15.54, respectively. The saponification value of the oil was 764.73 mg KOH/g. These values are very close that of Olaoye [19].

Fatty acid compositions of the PSO are also presented in Table 6. These results indicated that the PSO was highly saturated (87.5%) with the major fatty acids as lauric acid (10.49%), myristic acid (50.99%), palmitic acid (7.9%), 11-octadecenoic acid (10.96%) and stearic acid (1.8%). Methyl esters of the PSO will have better oxidative stability and fewer NOx emission but high cold flow properties as a result of highly saturated fatty acid composition [43].

Table 6. Physicochemical properties and fatty acid composition of PSO

Properties	Value	Limits (AOCS)	
Density (kg/m^3)	978.0	942	
Flash point (°C)	220	NS	
Acid value (mgKOH/g)	4.77	1.24	
%FFA	15.54	-	
Saponification value KOH/g	764.73	-	
Fatty acid composition	structure		
		PSO[a]	Pycnanthus seed oil[b]
Saturated			
Lauric	C13:0	15.86	64.72
Myristic	C15:0	50.99	4.33
Palmitic	C17: 0	7.88	13.97
11-octadecenoic	C19:0	10.96	ND
Stearic	C11:0	1.78	1.93
Unsaturated fatty			
Octadacenoic	C19:0	12.53	ND

[a]Present study; [b][19]; [c]Not specified; [d]Not detected

4. CONCLUSIONS

From the dimension/engineering and key fuel related properties of the oil resulting from the process extraction condition, the following conclusion can be deduced:

- Pycnanthus seeds possess geometrical shape which can be taken as ellipsoids. Hence, bio-data from the basic and complex geometric can be utilized for designing de-shelling equipment, oil screw expeller and in-situ batch reactor.
- An optimum yield (38.64%) was obtained at condition of 6.0 ml/g of hexane to seed ratio and extraction duration of 8 h.
- The fatty acid composition (FAC) of pycnanthus seed oil (PSO) analyzed by GS-MS technique was highly saturated (87.5%): lauric acid (10.48%), myristic acid (50.98%), palmitic acid (7.9%), 11-octadecanoic acid (10.96%) and stearic acid (1.8%). Based on the FAC, the methyl esters of PSO will exhibit better oxidation stability and fewer NOx emission but high cold flow properties.

» The basic fuel related properties of the PSO indicate that the oil can be explored for biofuel and industrial application

Acknowledgments

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