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Comparative Extraction of Some Non-Conventional Oil Seeds (Pentaclethra Macrophylla Benth) Using Different Solvents

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***Abstract*— Comparative extraction of some non-conventional oil seeds (Pentaclethra macrophylla benth) using n-hexane and ethanol have been investigated. Extraction parameters such as time, particle size and volume of solvent were varied and statistically analyzed. Also, the physicochemical properties of the oil extracted were studied in order to compare the extraction efficiency of the two solvents. The results of the physicochemical properties for n-hexane and ethanol are as follows; colour: raw sienne (dark brown) and raw umber (light brown), Density: 0.918g/cm^3 and 0.921g/cm^3 , melting point: 48 ^{0}C and 47^{0}C, Saponification value: 152.72 mg/KOH/g and 164.39 mg/KOH/g, iodine value: 52.51 mg/KOH/g and 49.95 mg/KOH/g , acid value: 5.84% and 6.40% , free fatty acid value: 2.92 % and 3.20% , refractive index: 1.453 and 1.466 at 25^{0}C and pH: 6.10 and 7.14, viscosity: 0.9100 and 0.9013 respectively. The coefficient of determination (R^2) value obtained from ANOVA showed that ethanol is a good alternative solvent to n-hexane.**

***Index Terms*— Extraction, Oil seeds, Solvent, Physicochemical and Anova**

I. INTRODUCTION

Generally, oils and fats from seeds and nuts constitute an essential part of man's diet. Fats and oils, together with proteins, carbohydrates, vitamins and minerals, are the main nutrients required by the human body. Fats and oils are rich sources of energy, containing two and a half times the calories of carbohydrates (per unit weight). In addition to being a source of vitamins A, D, E and K, fats and oils also contain essential fatty acids. These essential fatty acids are not manufactured by the body and must be obtained from diets, with linoleic, oleic and linolenic acids as examples of unsaturated fatty acids .

Vegetable oils are essential in meeting nutritional demands of the globe and are utilized for many food and industrial purposes .Plant seeds have been used since antiquity as sources of vegetable oil. Examples of some plant seeds which have been conventionally exploited commercially for this purpose includes soybeans, cotton seed, groundnut, corn, palm seeds and sunflower [1]. These conventional sources of vegetable oil are unable to meet the ever increasing demands of domestic and industrial sectors. Fixed oil derived from plants are usually obtained from plant seeds generally referred to as oil seeds. Conventional oilseeds are class of plants in which relatively large amount of lipids are stored in the seed tissue [2].There are wide varieties of seeds and nuts that can produce oils for food, pharmaceuticals, skincare products, aromatherapies, fuels and industrial lubricants. Also, some of these plant derived oils can be used to make soap, body and hair oils, detergents and paint. Vegetable oils derived from plant seeds have been playing vital roles to provide comfort in human lives in various aspects. Outside the realm of food manufacture, vegetable oils also feature in a variety of industrial uses ranging from the manufacturing of soap to the production of lubricants ,varnishes , plastics and paints. For instance, they have been used for illumination and lubricating purpose, production of detergents and cosmetics and for coatings and paint for many centuries before an abundant and cheap supply of mineral oil became available.

Nigeria, as a tropical country, has a wide variety of domestic plants that produce oil-bearing seeds of sufficient volume potential; for example, edible seeds like soya bean, peanuts, and corn. According to [3], Nigeria has one of the most extensive flora in continental Africa. Unfortunately, the vast majority of the seed oils have not been adequately study and characterized. A lot of work has been carried out on analysis of seed Oils by a number of researchers, primarily because of extensive demands for oils both for human consumption and for industrial applications; consequently there is an increasing need to search for oils from non-conventional sources to augment the available ones and also to meet specific applications. Among these non-conventional oil seeds includes Pentaclethra macrophylla benth (African oil bean seed), African star apple, African pear, vine rubber etc. Therefore, it is worth while to carried out extraction of oil from some non–conventional oil seed such as African oil bean seed.

However, continuous exploration and extraction of oils from the non– conventional oil seeds is expected to check the expensive prices of the conventional oils thus making them affordable to people, most especially in the developing countries [4].This work seek to compare the effects of different solvents on non-convectional oil seed (Pentacletra Macrophylla).

II. MATERIALS AND METHOD

A. Materials

Pentaclethra macrophylla benth used for this work were obtained from Ozoro market in Isoko North Local Government Area of Delta State of Nigeria.

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The solvents used were n- hexane and ethanol. All chemical used were of analytical grade and supply were made by Science Laboratory Technology Department of Delta State Polytechnic, Ozoro,.Nigeria

B. Preparation of sample

The sample seeds were thoroughly washed and seed coat were removed with sharp knife, the embryo(endoderm) were sliced into smaller pieces and dried in an oven at a temperature of about 105^{0}C - 110^{0}C. After cooling ,the sliced dried seeds were ground using a manual grinding machine and sieved through five different laboratory sets of sieves to obtain particle sizes of 0.85mm, 1.18mm, 2.36mm, 3.35mm and 6.70mm. The samples were stored in separate air tight containers and labeled adequately

C. Procedure

A known mass (say 20g) of milled sample of African oil been seed was weigh with Setra analytical weighing balance with model number BL- 410s and placed in the thimble of the Soxhlet extractor. A determined quantity of solvent was filled into the thimble of 250 ml Shuniu GG-17 Soxhlet extractor. The heating mantle was set at a specified temperature for the experiment and the extraction was carried out for a given (set) time. After the extraction with solvent, the set up (Soxhlet extractor) was dismantled and the miscella (mixture of solvent and extracted oil) obtained was poured into evaporation dish and placed on the heating mantle. The heating mantle was set at 78^{o}C and 68^{o}C for ethanol and n-hexane which is their boiling point respectively , so that , the solvent is brought to the vaporization point. After evaporation process, the weight of the oil was determined. This procedure was repeated for ethanol and n-hexane.for different experimental conditions. The oil extract was Characterized for the determination of oil quality by ASTM and AOCS(1993) methods.

III. RESULTS AND DISCUSSIONS

A. RESULTS

The results of the physico-chemical properties of the extracted oil for both solvents is presented in table 4.. Tables, 4.2, 4.3, 4.4 and figures 4.1, 4.2 and 4.3 presents result on the effects of particle size on oil yield, effects of time on oil yield and effects of solvent volume on oil yield respectively.

TABLE 4.1: PHYSICO-CHEMICAL PROPERTIES OF PENTACLETHRA MACROPHYLLA BENTH OILEXTRACTED WITH ETHANOL AND N-HEXANE

EXTRACTED OIL PROPERTIES	SOLVENTS	
	ETHANOL	N-HEXANE
Refractive index (25^{0}C)	1.453	1.466
Melting point (^{0}C)	48^{o}C	47^{o}C
Density (g/cm^3)	0.918	0.921
Viscosity (cP)	0.9100	0.9013
pH Value	6.10	7.14
Colour	Raw sienne(dark brown)	Raw umber (light brown)
Saponification value (meq/KOH/g)	152.72	164.39
Acid value (mg/KOH/g)	5.84	6.40
Iodine value (mg/100g of sample)	53.19	76.4
Free fatty acid (mg/KOH/g)	2.92	3.20

TABLE 4.2: EFFECTS OF PARTICLE SIZE ON OIL YIELDS

S/N		Extraction With N-Hexane		Extraction With Ethanol	
	Particle Size (mm)	Mass of Oil Extracted (g)	% Oil Yield	Mass of Oil Extracted (g)	% Oil Yield
1.	0.85	8.412	42.06	6.502	32.51
2.	1.18	7.800	39.00	6.105	30.53
3.	2.36	6.350	31.75	5.509	27.55
4.	3.35	6.088	30.45	5.157	25.79
5.	6.70	4.490	22.45	3.846	19.23

TABLE 4.3: EFFECTS OF TIME ON OIL YIELDS

S/N		Extraction With N-Hexane		Extraction With Ethanol	
	TIME (min.)	Mass of Oil Extracted (g)	% Oil Yield	Mass of Oil Extracted (g)	% Oil Yield
1.	20	7.003	35.02	5.912	29.56
2.	30	8.328	41.64	6.661	33.31
3.	40	8.566	42.83	6.793	33.97
4.	50	8.602	43.01	8.280	41.40
5.	60	8.763	43.82	8.824	44.12

TABLE 4.4: EFFECTS OF VOLUME OF SOLVENT ON OIL YIELDS

S/N	Volume of Solvent used (ml)	Extraction With N-Hexane		Extraction With Ethanol	
		Mass of Oil Extracted (g)	% Oil Yield	Mass of Oil Extracted (g)	% Oil Yield
1.	100	7.623	38.12	6.003	30.02
2.	125	9.258	46.29	7.976	39.88
3.	150	10.040	50.39	8.324	41.62
4.	175	10.478	52.39	8.829	44.15
5.	200	10.527	52.64	9.159	45.80

B. PHYSICOCHEMICAL PROPERTIES

Table 4.1 presents the respective physicochemical properties of the extracted oil of Pentaclethra macrophylla benth. For the physical properties, the oil extracted is raw sienne (dark brown) with ethanol and raw umber(light brown) with n-hexane. The result is consistence with the one reported by [5] and [6]). The refractive index for n-hexane and ethanol extract of the oil are 1.466 and 1.453 respectively. The refractive index indicates the level of optical charity of the crude oil sample in relative to water. The refractive index of oil is a measure of how much a light ray is bent when if passes from air into oil and is usually depends on the density of the oil. This result was consistence to the 1.46 value obtained at 25^{o}C for oil bean by [7]; and 1.4672 obtained by [8] on Chrysophyllum albidum seed oil. And these result show that, the oil is not as thick as most drying oils whose refractive indices fall between 1.475 and 1.485 [9]. Also the physical analysis of the oil gave a pH of 6.10 and 7.14 respectively for ethanol extract and n- hexane. The result indicate that, the ethanol extract is almost acidic in nature while n-hexane extract is almost neutral. The melting point for oil extract were 48^{o}C and 49^{o}C respectively for ethanol and n-hexane. This values are higher than the 22^{o}C±1.02 reported by [6]. This results is an indication of the fluidity of the oil. The viscosity of the oil extract shows 0.9100cP and 0.9113cP respectively for ethanol and n-hexane. This low value of viscosity of the oil shows that , the oil has low resistant to shear force(stress). The density obtained for the oil were 0.918g/cm^3 and 0.921g/cm^{3}for ethanol extract and n- hexane extract respectively. These results is an indication that the oil is less dense than water and the result is comparable to 0.9111 value obtained by [10] on fluted pumpkin oil.

And for the chemical analysis, the results shows that the oil obtained has low iodine value of 53.19mg iodine/100g of sample and 76.4mg iodine/100g of sample for ethanol and n-hexane respectively. Since iodine value of the Pentaclethra macrophylla benth oil is lower than 100, it can be classified as a non-drying oil. This means that, the oil has a low content of unsaturated fatty acids. Iodine value is very important because it help in the estimation of the individual fatty acid content of the oil [11]. And because of the low value of the iodine value, the oil cannot be used in varnish, coating and paint industries. It could be suitable as lubricating oil and for soft soap and candle production.

The free fatty acid obtained from the chemical analysis were 2.92 for ethanol extract and 3.20 for n-hexane extract. This values agrees with the 2.25 obtained by [8] on Chrysophyllum albidum seed oil. Since the free fatty acid of Pentaclethra macrophylla oil obtained is low, this suggest its usage as

edible oil. Also the acid value results shows 5.84mg/KOH/g) and 6.40mg/KOH/g) respectively for ethanol and n- hexane extract respectively. This result is comparable to the 7.35±0.12 and 7 acid value reported by [6].. The acid value is a measure of freshness and edibility of the oil. And for the saponification value, the result indicates 152.72 and 164.39 respectively for ethanol and n-hexane extract. [5] and [10] reported a saponification value of 158.40±3.40 and 162.69mg/KOH/g respectively on some non conventional oil. The high saponification value recorded for the oil is an indication that , the oil have potential for industrial used [12].

C. EFFECT OF PARTICLE SIZE ON OIL YIELD

The results of effect of particle size on oil yield is shown in table 4.2. The extraction was carried out using five different particle size of 0.85mm, 1.18mm, 2.36mm, 3.35mm and 6.70mm for both solvents. It was observed that as particle size decreases ,oil yield increased for both solvents. The highest yield was noted when the particle size was 0.85mm with a percentage yield of 42.06 and 32.51 respectively for n-hexane and ethanol . Beyond this particle size , the oil yield decreases for both solvent as shown in figure 4.1. In addition, n- hexane extract a little more of the oil than ethanol in all cases of particle size used for this experiment.

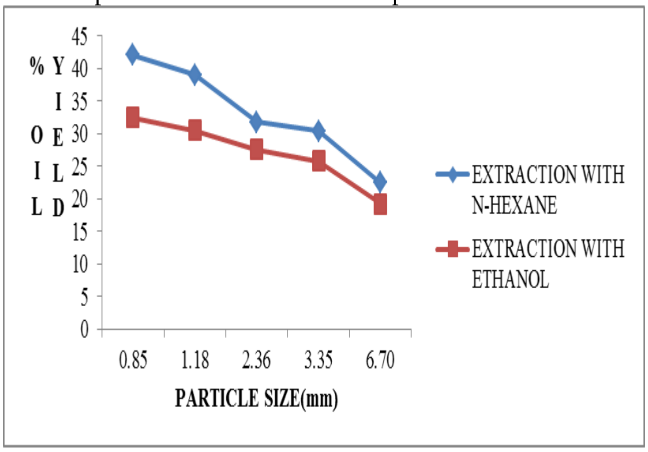

FIGURE 4.1: EFFECT OF PARTICLE SIZE ON OIL YIELD

The low oil yield experienced for larger (bigger) particle sizes is as a result of lesser surface area , thereby restricting the penetration of solvent into the core of the seed to leach the oil out of the seed. But the high yield experience in smaller particles is attributed to the large surface area of the particles which enable the solvent to penetrate into the core of the seed to leach the oil out of the seed. Large particles have smaller amount of surface areas and are more resistant to intrusions of solvent and oil diffusion. Therefore , small amount of oil will be carried from inside the large particles to the surrounding solution.

D. EFFECTS OF TIME ON OIL YIELD

The extraction time is also called residence time or contact time. This is very important in solvent extraction of oil because it helps in choosing the most optimal time of extraction. In this research work, extraction time of 20,30,40,50,and 60 minutes was used. The result is shown in table 4.3 and also presented in figure 4.2.

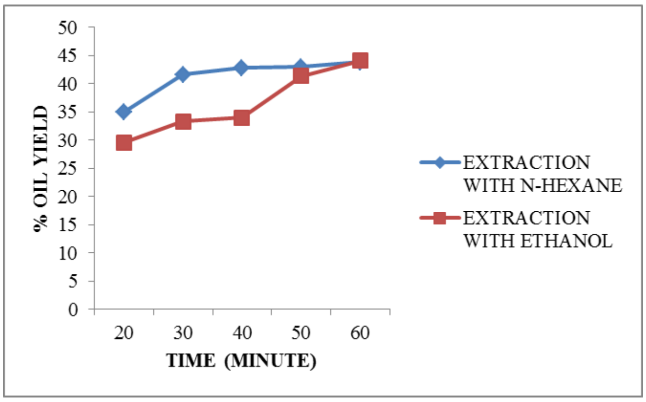

FIGURE 4.2: EFFECTS OF TIME ON OIL YIELD

STATISTICAL ANALYSIS:

IV. EFFECTS OF PARTICLE SIZE ON OIL YIELDS

SUMMARY OUTPUT FOR EXTRACTION WITH N-HEXANE

Regression Statistics	
Multiple R	0.959
R Square	0.919681
Adjusted R Square	0.892908
Standard Error	2.520832
Observations	5

ANOVA

	Df	*SS*	*MS*	*F*	*Significance F*
Regression	1	218.2865	218.2865	34.35098	0.009904
Residual	3	19.06378	6.354594		
Total	4	237.3503			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	42.21959	1.915663	22.03916	0.000204	36.1231	48.31609
Particle Size (mm)	-3.14321	0.536295	-5.86097	0.009904	-4.84994	-1.43648

RESIDUAL OUTPUT

Observation	*Predicted Extraction With N-Hexane (% Oil Yield)*	*Residuals*
1	39.54786	2.512136
2	38.5106	0.489396
3	34.80162	-3.05162
4	31.68984	-1.23984
5	21.16008	1.28992

SUMMARY OUTPUT EXTRACTION WITH ETHANOL

Regression Statistics	
Multiple R	0.991443
R Square	0.98296
Adjusted R Square	0.977279
Standard Error	0.77186
Observations	5

ANOVA

	Df	*SS*	*MS*	*F*	*Significance F*
Regression	1	103.0988	103.0988	173.052	0.000949
Residual	3	1.787303	0.595768		
Total	4	104.8861			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	33.36056	0.586562	56.87477	1.2E-05	31.49385	35.22726
Particle Size (mm)	-2.16016	0.16421	-13.1549	0.000949	-2.68275	-1.63758

RESIDUAL OUTPUT

Observation	*Predicted Extraction With Ethanol (% Oil Yield)*	*Residuals*
1	31.52442	0.985585
2	30.81156	-0.28156
3	28.26257	-0.71257
4	26.124	-0.334
5	18.88745	0.342547

2: EFFECTS OF TIME ON OIL YIELDS
SUMMARY OUTPUT FOR EXTRACTION WITH N-HEXANE

Regression Statistics	
Multiple R	0.838667
R Square	0.703363
Adjusted R Square	0.604484
Standard Error	2.249209

Observations	5

ANOVA

	Df	*SS*	*MS*	*F*	*Significance F*
Regression	1	35.98609	35.98609	7.113361	0.075878
Residual	3	15.17683	5.058943		
Total	4	51.16292			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	33.676	3.017631	11.15975	0.001542	24.07255	43.27945
Time (min.)	0.1897	0.071126	2.667088	0.075878	-0.03666	0.416055

RESIDUAL OUTPUT

Observation	*Predicted Extraction With N-Hexane (% Oil Yield)*	*Residuals*
1	37.47	-2.45
2	39.367	2.273
3	41.264	1.566
4	43.161	-0.151
5	45.058	-1.238

SUMMARY OUTPUT EXTRACTION WITH ETHANOL

Regression Statistics	
Multiple R	0.971136
R Square	0.943106
Adjusted R Square	0.924141
Standard Error	1.668599
Observations	5

ANOVA

	Df	*SS*	*MS*	*F*	*Significance F*
Regression	1	138.4584	138.4584	49.72963	0.005861
Residual	3	8.35267	2.784223		
Total	4	146.8111			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	21.588	2.238661	9.643265	0.002367	14.46358	28.71242
Time (min.)	0.3721	0.052766	7.051924	0.005861	0.204176	0.540024

RESIDUAL OUTPUT

Observation	*Predicted Extraction With Ethanol (% Oil Yield)*	*Residuals*
1	29.03	0.53
2	32.751	0.559
3	36.472	-2.502
4	40.193	1.207
5	43.914	0.206

3: EFFECTS OF VOLUME OF SOLVENT ON OIL YIELDS
SUMMARY OUTPUT FOR EXTRACTION WITH N-HEXANE

Regression Statistics	
Multiple R	0.916377
R Square	0.839748
Adjusted R Square	0.78633
Standard Error	2.802651
Observations	5

ANOVA

	Df	*SS*	*MS*	*F*	*Significance F*
Regression	1	123.482	123.482	15.72047	0.028661
Residual	3	23.56456	7.854853		
Total	4	147.0465			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	26.882	5.463373	4.920404	0.016084	9.495109	44.26889
Volume of solvent used (ml)	0.14056	0.035451	3.964904	0.028661	0.027739	0.253381

RESIDUAL OUTPUT

Observation	*Predicted Extraction With N-Hexane (% Oil Yield)*	*Residuals*
1	40.938	-2.818
2	44.452	1.838
3	47.966	2.424
4	51.48	0.91
5	54.994	-2.354

SUMMARY OUTPUT EXTRACTION WITH ETHANOL

Regression Statistics	
Multiple R	0.917003
R Square	0.840894
Adjusted R Square	0.787859
Standard Error	2.845501
Observations	5

ANOVA

	Df	*SS*	*MS*	*F*	*Significance F*
Regression	1	128.3789	128.3789	15.85536	0.028343
Residual	3	24.29063	8.096877		
Total	4	152.6695			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	18.796	5.546903	3.388558	0.04282	1.143279	36.44872
Volume of solvent used (ml)	0.14332	0.035993	3.981879	0.028343	0.028774	0.257866

RESIDUAL OUTPUT

Observation	*Predicted Extraction With Ethanol (% Oil Yield)*	*Residuals*
1	33.128	-3.108
2	36.711	3.169
3	40.294	1.326
4	43.877	0.273
5	47.46	-1.66

Oil yield increased as the extraction time increased for n-hexane and ethanol. The oil yield were seen increasing in the first 20 minutes of extraction and thereafter the yield of oil started reducing. As time increases from 20 to 60 minutes, oil yield increases from 35.02% to 43.82% and from 29.56% to 42.12% respectively for n-hexane and ethanol. The highest oil yield recorded was at 60 minutes, though values were almost insignificant. The initial high rate of extraction may be due to quick solubility of the oil present at the solid surface and higher mass transfer driving force provided by low oil concentration in fresh solvent. The later slower rate may be attributed to lower driving force due to increasing oil concentration in the solvent .

4.5: EFFECTS OF VOLUME OF SOLVENT ON OIL YIELDS

The effects of volume of solvent on oil yield is shown in table 4.4 and also in figure 4.3**.** The experiment was carried out using 1.18mm particle size of sample for 20 minutes each with the following volumes- 100, 125, 150, 175 and 200 ml. From the result, it was found that oil yield increased as the volume of solvent increased from 100- 200ml.

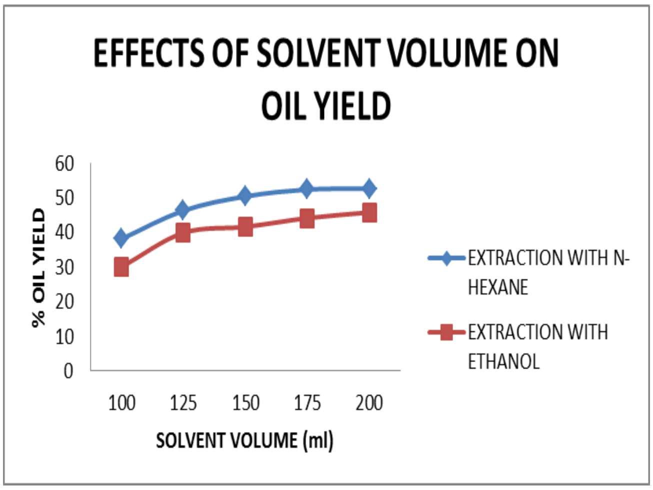

Figure 4.3: EFFECTS OF SOLVENT VOLUME ON OIL YIELD

the oil yield increases with volume of solvent up to 150ml, then the yield becomes less and insignificant as extraction progresses. This was been reported by [9] in his studied of kinetics and thermodynamics study of oil extraction from fluted pumpkin seed. Also [11] reported that, the positive effect of volume of solvent on oil yield was as a result of increase in the concentration driving force as volume of solvent increases. It was as a result of increased washing of oil extracted away from the particle surface by the solvent as a result of increased volume. The highest percentage of oil yield were attained when a solvent volume of 200ml was used. But this was insignificant compare to when 150ml was used.

V. CONCLUSION

It can be concluded that ethanol, a green and safe solvent is a good replacement for n-hexane statistically. There is no significant difference in the extraction efficiencies of n-hexane and ethanol based on experimental parameters such as time, volume of solvent and particle size affect the oil yield. The oil properties reveal that the types of solvent has influence over the oil properties with variance. Different solvents will yield different extracts and extract compositions. Finally, non-conventional oil seeds such Pentaclethra macrophylla benth has great potential for oil that could replace the conventional oil seeds industrially for the production of soaps, candles, shampoo and the oil can be regarded as edible.

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