

Solid-liquid extraction of oils of African elemi's (*Canarium schweinfurthii*'s) fruit

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Abstract: African elemi (*Canarium schweinfurthii*) contains oil in its fruit's pulp and kernel. Solvent extraction of these oils using n-hexane was investigated in this work to obtain some process parameters. This was done at different temperatures (30°C - 55°C) over a period of time (30 - 240 min) with varying solvent to feed (solid) mass ratios. Oil yield and extraction efficiency were observed to increase with time, temperature and materials ratio but in different fashions. The kinetics of the oils' extraction was found to be adequately described by Peleg equation and a variant of it. At 30°C, kernel oil recorded the highest saturated extraction yield (C_s) with 7.5 g solvent/g solid while no peak was observed for pulp oil extraction using 1 to 10 g solvent/g solid. Kernel oil gave the highest saturated (maximum) yield (45.93 g/100 g solid) but pulp oil gave the highest initial extraction rate ($B_0=3.5868$ g/L.min) and fastest time constant ($\tau_0=9.84$ min). Free fatty acid content of the extracted oils was observed to increase with leaching temperature while the iodine value reduced with it. This phenomenon was more pronounced with kernel oil.

Keywords: African elemi fruit, solvent extraction, pulp and kernel oils, extraction kinetics, Peleg equation

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1 Introduction

African elemi (*Canarium schweinfurthii*) is one of the tropical trees whose fruits contain oils in its pulp and seed kernel. The tree is grown widely in the tropics and is abundantly available in Sub-Sahara Africa, including Nigeria (Burkill, 1994; Keay, 1989; Orwa et al., 2009, Hafchinson and Dalziel, 1954). The pulp is commonly eaten raw or cooked; it is also usually processed for the constituent oil which is popularly referred to as *atile* oil in some parts of northern Nigeria. The seed (stone) which contains the kernel oil is either thrown away or used as local beads for feet (Burkill, 1994).

The properties and qualities of the two oils have been investigated to some extent (Danjouma et al., 2006; Abayeh, Abdulrazaq and Olaogun, 1999; Georges, Olivier and Simard, 1992) and the oils have been shown to have potential industrial uses in production of pharmaceutical and personal products and, as a thermal fluid among others (Daniel, 1965; Apple-White, 1980; Eromosele et al., 1998; Ajiwe et al., 1998). In addition, researches on the processing of the fruit for oil recovery have been reported (Tchiegang et al., 2004a, 2004b). However, unlike some other oil-bearing materials such as groundnut, cotton seed, soyabean, palm pulp and palm kernel, the extraction of oils from elemi pulp and kernel are not being carried out at commercial level at present, despite ready availability of the fruit in large quantity in Nigeria and elsewhere in Sub-Sahara Africa. This situation would improve if data needed for the design and operation of the oils' extraction plants are available.

The oils and fats in vegetable oil-bearing materials are usually located within the cells of the materials. The morphology of oil bearing material determines, to a large extent, the mode and ease of oil extraction from these materials (Daniel, 1965; Richardson and Harker, 2002; Hanim, 1992; McCabe, Smith and Harriot, 1984). Though many studies have been reported on solid-liquid extraction of vegetable oils and allied products, the leaching parameters are known to be different for different materials largely because of differences in the cellular structures of oil bearing materials (Sayyar et al., 2009; Franco et al., 2007; Meziane and Kadi, 2008; So and Macdonald, 1986; Bucic-Kojic et al., 2007).

In this study, effects of temperature on oil extraction yield and efficiency and, on some physico-chemical properties of oils extracted from African elemi's fruit were studied. Extraction kinetics of the oils were established at 30°C with solvent to solid mass ratios between 1:1 and 1:10, using n-hexane on pulp and kernel particle sizes less than 2 mm.

2 Materials and methods

Pulp and kernel of the fruit of African elemi tree were leached under different conditions of temperatures, solvent-solid ratio and time using n-hexane as the solvent. Some of the oil samples obtained were thereafter subjected to physico-chemical analysis.

2.1 Sample preparation

Fresh fruits of African elemi were obtained from a market in Nigeria and were cleansed of extraneous materials. The fruits were then de-pulped and the pulps were oven-dried at 40°C to an equilibrium moisture content of 9.90%. The seeds (stones) left after de-pulping the fruits were cracked to yield the kernels which were dried to equilibrium moisture content of 5.03%.

The dried pulps and kernels were thereafter grounded to particles of less than 2 mm size using Retsch KG grinder. These particles were used in subsequent analysis and leaching experiments. The total fat and oil contents of the prepared samples were determined by digesting with 4N HCl as described by Jacobs (1982).

2.2 Batch solvent extraction of pulp and kernel oils

A quantity of 50 g of each of the prepared samples (pulp and kernel particles) was added with 76 ml n-hexane (corresponding to 1:1 solvent-solid mass ratio) in 500 ml round bottom flask. The mixture was then subjected to constant agitation at 30°C for one hour and thereafter filtered to give the filtrate (miscella) and the cake. The miscella was then heated at 40°C to recover the solvent for reuse and the mass of the remaining oil was recorded. This leaching experiment was repeated at 35°C, 40°C, 45°C, 50°C and 55°C for solid to solvent ratios of 1:1, 1:2 and 1:4. The oil yield and extraction efficiency were calculated using Equations 1 and 2 respectively.

$$\text{Yield} = \left(\frac{\text{Mass of oil}}{\text{Mass of particles}} \right) 100 \quad (1)$$

$$\text{Efficiency} = \left(\frac{\text{Mass of oil extracted}}{\text{Mass of oil in particles}} \right) 100 \quad (2)$$

By following similar steps as described earlier, the prepared materials were subjected to extraction by n-hexane using solvent to solid mass ratios of 1:1, 1:2.5, 1:5, 1:7.5 and 1:10 for 0.5, 1, 2 and 4 hours at 30°C. The oil yield and extraction efficiency were obtained as described previously.

2.3 Determination of properties of pulp and kernel oils

Acid, iodine, and saponification values as well as unsaponifiable matters and specific gravity of the oil samples were determined using the standard methods (Jacobs, 1982; Pearson, 1976). Acid and iodine values were obtained for oil extracted in one hour at six temperatures between 30°C and 55°C while the rest properties were determined for extracts obtained at 30°C.

3 Results and discussion

3.1 Properties of pulp and kernel oils

The total oil content of the pulp was determined as 49.92 ± 1.42 wt% (dry basis). The saponification value, unsaponifiable matters, and specific gravity of the pulp oil extracted at 30°C were obtained as 191.44, 1.15% and 0.9291 respectively. These physicochemical properties' values are not far from the typical values (Burkill, 1994; Hafchinson and Dalziel, 1954). A cursory assessment shows that temperature does not have significant effects on acid and iodine values of the extracted pulp oil (Table 1). An increment of 0.0207 meq KOH/g oil and a decrease of 1.826 in acid and iodine values respectively, were observed between 30°C and 55°C.

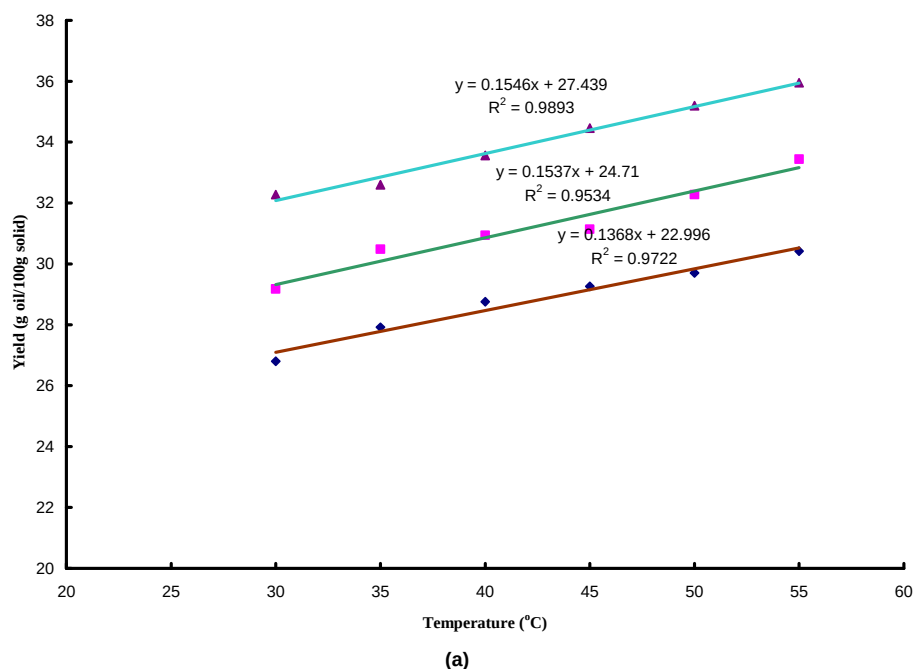
The oil content of kernel oil was 51.79 ± 1.20 wt% (dry basis). The saponification value, unsaponifiable matter and specific gravity of the oil extracted at 30°C were 193.545, 1.745% and 0.9143 respectively. Acid values changed by 0.098 meq KOH/g oil between 30°C and 55°C while analogous change for iodine value was 2.7918. The change in acid value of about 10% is substantial and far higher than the change recorded for pulp oil. The same trend was observed for change in iodine values (Table 1).

Table 1 Temperature effects on properties of extracted pulp and kernel oils

Properties		Values at different temperatures					
		($^{\circ}\text{C}$)					
		30	35	40	45	50	55
Acid value (meq KOH/g oil)	Pulp	4.0159	4.0303	4.0305	4.0324	4.0364	4.0366
	Kernel	0.9818	0.9858	1.0379	1.0519	1.0696	1.0799
Free fatty acid (%)	Pulp	2.0200	2.0273	2.0273	2.0283	2.0304	2.0304
	Kernel	0.4984	0.5089	0.5220	0.5291	0.5361	0.5432
Iodine value	Pulp	87.381	87.297	87.138	86.826	86.523	86.278
	Kernel	94.160	93.145	92.891	92.383	91.622	91.368

3.2 Pulp and kernel oil yield and extraction efficiency

The oil yield (and thus extraction efficiency) for pulp and kernel oils (Figure 1a, b) were observed to increase with temperature and solvent-solid ratios. The variation of the two parameters with temperature is fairly linear. Thus the extract obtained at 60 min with 1:4 mass ratio gave the highest oil yield and extraction efficiency at 55°C for the two oils. Figure 1 Extraction yield against temperature with different solvent mass for (a) pulp oil (b) kernel oil



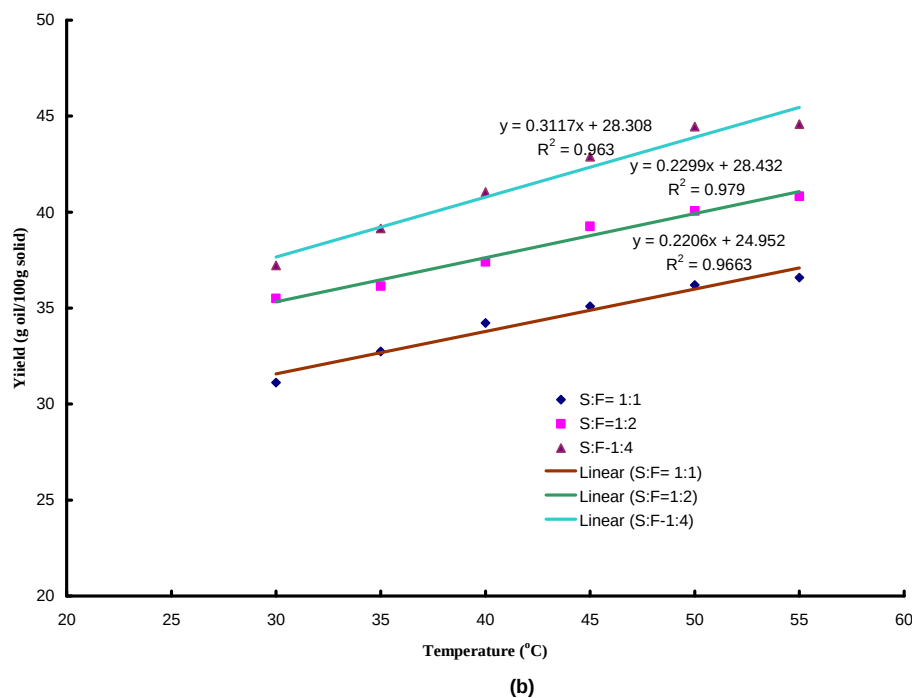


Figure 1 Extraction yield against temperature with different solvent mass for (a) pulp oil (b) kernel oil.

For pulp oil, the maximum oil yield and extraction efficiency were 35.26 and 70.63% respectively. For kernel oil, analogous values were 45.58 and 79.15%.

The trend observed in variation of yield and efficiency with temperature and mass (or volume) of solvent used, is expected. In general, viscosity reduces with the temperature and, lower viscosity improves diffusivity which enhances the transport of the liquid in and out of the pores of the oil-bearing materials. In addition, the solubility of oil in hydrocarbons such as n-hexane is expected to increase with the temperature and volume of the solvent and so is the yield and extraction efficiency (Hanim, 1992; McCabe, Smith and Harriot, 1984; Sayyar et al., 2009; Franco et al., 2007).

The time rates of oil yield and extraction efficiency showed exponential decay trend (Figures 2 and 3), with the highest rate recorded for materials ratio of 10:1 for the extraction carried out at 30°C. Similar results have been obtained in related works (Sayyar et al., 2009; Franco et al., 2007; Meziane and Kadi, 2008; So and Macdonald, 1986; Bucic-Kojic et al., 2007). The optimum material ratio for kernel oil at 30°C was 7.5:1 as this ratio gave about the same yield and efficiency as 10:1. For pulp oil the optimum ratio at 30°C was not attained though 10:1 materials ratio produced the highest yield and extraction efficiency. The observed differences in the amount of solvent required for maximum extraction at 30°C between the pulp and kernel might be due to the differences in the cellular structures of the two materials (Hanim, 1992).

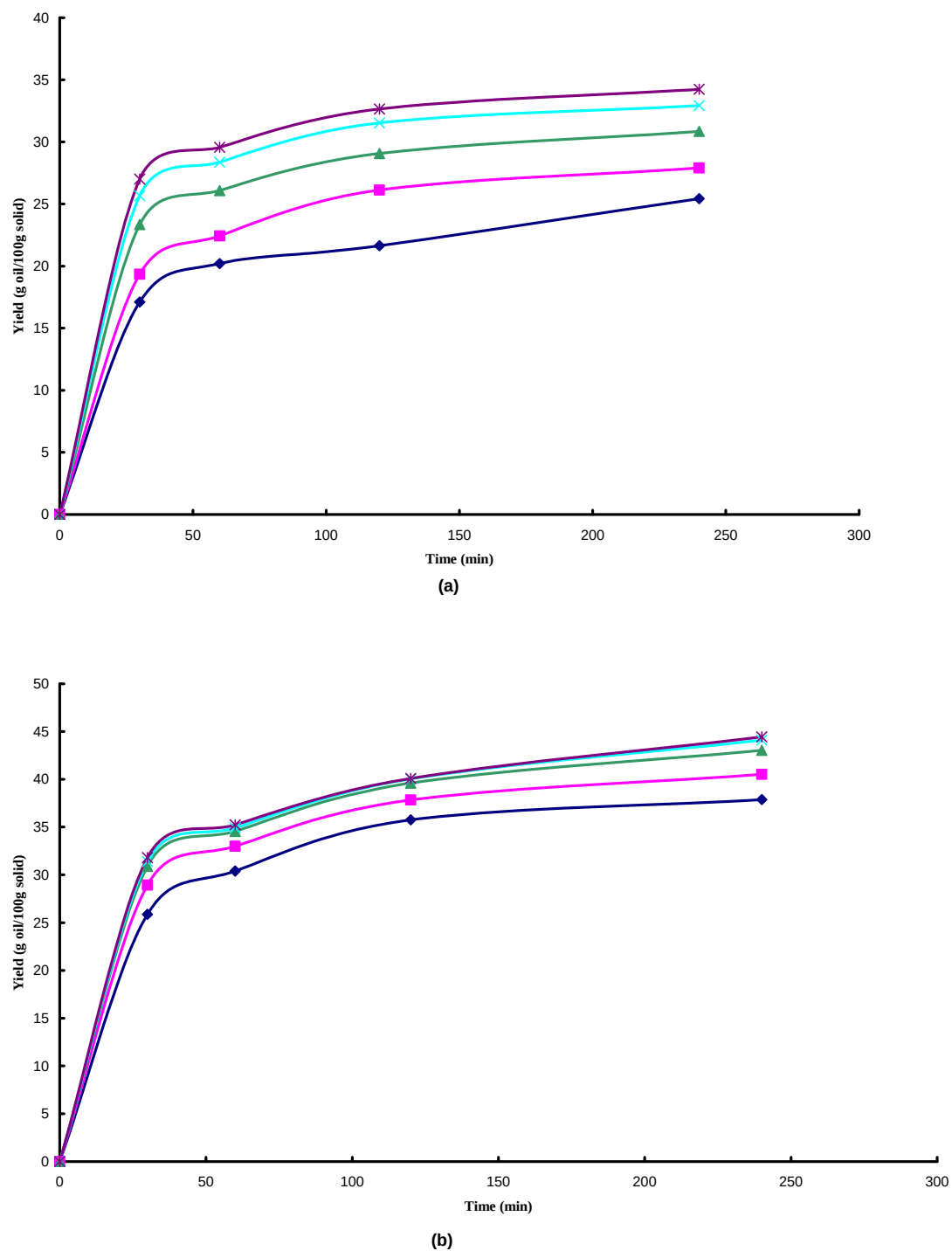


Figure 2 Extraction yield against time with different solvent mass for (a) pulp oil (b) kernel oil

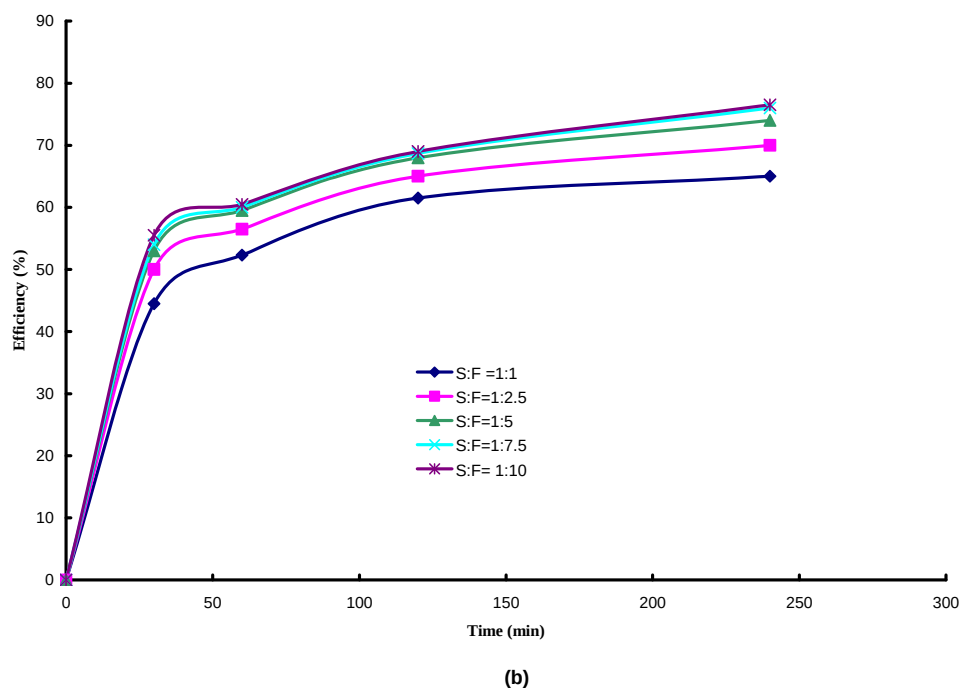
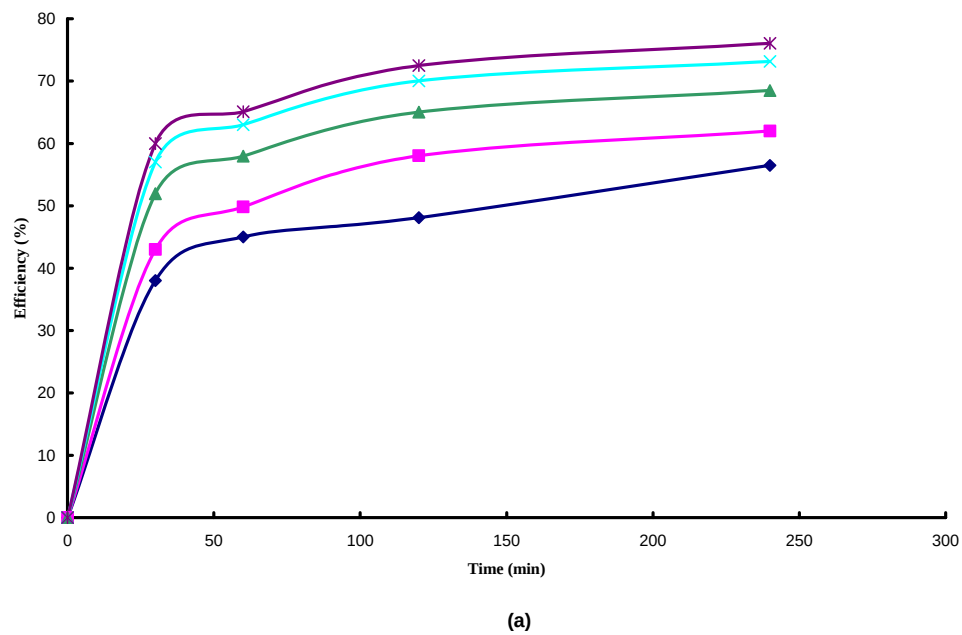


Figure 3 Extraction efficiency against time with different solvent mass for (a) pulp oil (b) kernel oil

3.3 Extraction kinetics of pulp and kernel oils

The time rate (kinetics) of extraction pulp and kernel oils was evaluated with DataFit 9 software. Equations 3 and 4 were found to be the most appropriate kinetic models for the oils with solvent masses between 1 and 10 g/g solid at 30°C.

$$C_t = t/(K_1 + K_2 t) \quad (3)$$

$$C_t = K'_1 t / (t + K'_2) \quad (4)$$

K_1 and K_2 are the Peleg rate constant (g solid.min/cg extract) and maximum capacity (g solid/cg extract) respectively (Meziane and Kadi, 2008; So and Macdonald, 1986; Bucic-Kojic et al., 2007). These parameters are the reciprocals of initial rate of extraction (B_0) and maximum extraction capacity (C_s) respectively. K'_1 is the maximum extraction capacity (C_s) and K'_2 is extraction time constant (τ_0 , min). The parameters' values are presented in Tables 2 and 3.

Table 2 Extraction kinetic models' parameters of oils of African elemi's fruit

Equation	Mass ratio	Pulp oil			Kernel oil		
		Parameters		R^2	Parameters		R^2
		K_1 or K'_1	K_2 or K'_2		K_1 or K'_1	K_2 or K'_2	
Equation 3	1:1	0.6533	0.03824	0.9928	0.4427	0.02459	0.9988
	2.5:1	0.5744	0.03368	0.9986	0.3569	0.02345	0.9985
	5:1	0.3700	0.03124	0.9989	0.3327	0.02229	0.9971
	7.5:1	0.3072	0.02928	0.9991	0.3350	0.02184	0.9959
	10:1	0.2788	0.02832	0.9989	0.3250	0.02177	0.9954
Equation 4	1:1	26.1520	17.0852	0.9928	40.6697	18.0041	0.9988
	2.5:1	29.6876	17.0526	0.9986	42.6432	15.2201	0.9985
	5:1	32.0083	11.8437	0.9989	44.8692	14.9303	0.9971
	7.5:1	34.1356	10.4868	0.9991	45.7903	15.3420	0.9959
	10:1	35.2963	9.8403	0.9989	45.9257	14.9295	0.9954

Table 3 Extraction process parameters of oils of African elemi's fruit

Oil	Mass ratio	Extraction process parameters		
		Maximum yield/ C_s (cg/g)	Initial rate/ B_0 (cg/g.min)	Time constant/ τ_0 (min)
Pulp Oil	1:1	26.1520	1.5307	17.0852
	2.5:1	29.6878	1.7409	17.0526
	5:1	32.0053	2.7027	11.8437
	7.5:1	34.1350	3.2552	10.4868
	10:1	35.2963	3.5868	9.8403
Kernel Oil	1:1	40.6697	2.2584	18.0041
	2.5:1	42.6432	2.8019	15.2201
	5:1	44.8692	3.0057	14.9303
	7.5:1	45.7903	2.9853	15.3420
	10:1	45.9357	3.0769	14.9294

The values of the coefficient of determination (R^2) indicate the appropriateness of the equations and their similarity. In Table 3, it can be seen that the maximum yield and initial rate increased with the solvent mass utilized while reversed relationship holds for time constant (Richardson and Harker, 2002; Hanim, 1992; McCabe, Smith and Harriot, 1984). This is to be expected as utilization of greater solvent mass leads to less viscous liquid which enhances the diffusion and extraction rate and reduces the time required for optimum extraction yield (time constant).

Though maximum yield is higher for kernel oil extraction, more time is required to achieve this compared to pulp oil extraction when the same solvent mass was employed. The yield gradient appeared to be greater with pulp than kernel oil extraction. This might be due to higher solubility of kernel oil in n-hexane but greater diffusion resistance of its miscella within the cellular structure of the kernel particles when compared to pulp oil and its miscella. Kernel oil achieved the peak of extraction yield using n-hexane with 7.5 g solvent/g solid while pulp oil extraction did not reach the peak with this mass ratio. Rather initial rate was seen to increase from 1.5307 to 3.5868 g oil/(100 g solid.min) for pulp oil as against 2.2584 to 3.0769 g oil/(g solid.min) for kernel oil. It thus appears that the solubility of the oils in the solvent and resistance to diffusion experienced by the miscella are responsible for the observed phenomenon.

4 Conclusions

The oil yield and extraction efficiency of pulp and kernel oil of elemi's fruit was found to increase linearly with temperature. These parameters were also found to increase with the volume of solvent used for extraction. A solid to solvent ratio of 1:7.5 was observed to be optimum for kernel oil extraction at 30°C while no optimum ratio was obtained for pulp oil under the same condition. Temperature change has greater effects on acid and iodine values in kernel oil than pulp oil while extracting with n-hexane.

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