Extraction and Characterization of Oil from Acacia Nilotica Seeds by Different Solvents

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Abstract: This study is aimed at studying the efficacy of three different solvents in the extraction of oil from Acacia Nilotica. The solvents employed are Petroleum Ether-Ethanol 50:50; Acetone and Chloroform-Methanol 50:50. The extraction on each of the solvents was done at different time interval of 2 h for five runs. The results obtained showed that the yield of oil when petroleum-ethanol (50:50) was used at 2,4,6,8 and 10 h are 1.71, 3.83, 5.82, 7.08 and 7.31%, respectively. The yield of oil obtained using acetone as solvent at the same time intervals are 5.37, 6.96, 7.71, 8.45 and 8.24% while for chloroform-methanol (50:50), the following yield of oil was obtained 1.37, 3.83, 6.58, 7.94 and 8.33% also at the same time interval. Findings revealed that chloroform- methanol mixture is a better solvent among the three used in the extraction. The result also alluded to the usual convention of extraction of oil increase with time.

Key words: Oil, extraction, acacia nilotica, ethene-ethanol, acetone, chloroform methanol

INTRODUCTION

The world demand for vegetable oil is constantly increasing due to increase in the world population. The production of vegetable oils and fats, which is around 30 metric tones, is not enough to meet the needs of people, since fats and oil are required industrially for the manufacturing of soap and other industrial purposes (Weiss, 2000; Gavriel, 1996). This research is part of the efforts of finding ways of increasing fats and oil production and it is shown by considering the extraction of oil from acacia seeds.

Acacia Nilotica is a tree of 5-20 m height with a dense spherical crown stems and branchlets usually dark to black colored, fissured back, grey-pinkish Flask, exuding a reddish low quality gum. The tree has thin, straight, grey spines in auxiliary pairs, usually in 3 to 12 pairs, 5 to 7.5 cm long in young trees, mature trees commonly without thorns. The leaves are bipinnate, with 3-6 pairs of pinnulate. Flowers in globulous heads 1.2 to 1.5cm in diameter of a bright golden-yellow colour. The tree has scented thorns all over the sterm and branches and are found mostly in riverine areas and seasonal flooded areas (Weiss, 2000; Macreie *et al.*, 1993; Paterson, 1989; Alfred and Patrick, 1985).

Acacia Nilotica seed contains protein, fat, nitrogen freed extract, Acid Detergent Fibre ADF, Crude Fibre CF, organic matter digestibility, metabolisable energy, Tannin, phosphorus, calcium, magnesium, potassium, silicon, sulphur, chlorine, copper, zinc, manganese and iron. The

seed has numerous chemical components and it finds its place in pharmaceutical industries. Bullocks fed 45% oilextract seeds of Acacia Nilotica in their diet showed reduced weight gain (68g day to 16g day). Acacia Nilotica tannin have been used to treat cotton seed cake to prevent rumen degradation of protein. Acacia Nilotica seed is made up of shell and kernel. The kernel finds many uses. It can be used as fuel and to make products like flour, starch, and cattle/poultry feed other than oil. The kernel yields good manure for plant. The fat in the kernel is edible and it can be substituted for cocoa-butter. Also, the fat and oil can be used industrially in manufacturing of antiseptic soaps (Macreie *et al.*, 1993; Paterson, 1989; Alfred and Patrick, 1985).

MATERIALS AND METHODS

In the extraction of oil from seed, two stages are involved: The preparatory stage and extraction stage. The preparatory stage include; separation of seeds from fruits, drying of seeds, size reduction of seeds, separation of shell from kernel, weighing and packaging of the kernel. The acacia fruits were collected from the tree, dried by the means of sunlight and pounded using pistol and mortal. The seeds were carefully separated from the pounded dried fruit. Then the seeds were exposed to sunlight for further drying. An electric grinding machine was used to grind the seed for two reasons. Firstly, to create enablement for the separation of the shell from the kernel. And secondly, for size reduction of the kernel to enhance

the extraction process. Then a sieve was used to separate the shell from the kernel. The kernel in a powder form passed through the sieve, while the shell was trapped at the top of the sieve. 5.00 g of the kernel in powered form was weighed using electric balance. Care was taken to prevent external factors, such as vibration, breeze and temperature to affect the measurement. Filter paper was used to package the sample (Kernel), since the filter paper serve as absorbent.

The extraction stage involves the use of the following equipments: soxhlet extractor, condenser, heating mantle, thimble, round bottom flask and filter paper. The soxhlet is a laboratory extraction apparatus that operates on the continuous process principles. The extraction apparatus was mounted on a heating mantle using a clamp. Five of such apparatus were arranged in series with water connection going in and out of the condensers. The sample was kept in the thimble and dropped into the soxhlet tube. The solvent was placed in the round bottom flask and the solvent evaporates passing through the tube into the soxhlet extractor. The vapour was then condensed by flow under gravity and percolates through the beds of the sample to extract the oil. As the condensation process continuous, the level of miscela in the tube rises until it reaches the upper part of the tube, then the miscella over flows back into the round bottom flask. In this way, the sample is continuously treated with freshly condensed solvent. At the end of the extraction, the sample was removed from the apparatus and dried using an electric oven and then re-weighed. The solvent was recovered from the oil using the same soxhlet apparatus by distillation (Perry and Green, 1997; Schweitzer, 1988; Fernando and Akuyobi, 1987).

For the determination of the oil content of the seed, 5.00 g each of the sample were put in a filter paper and the weight of the sample and filter paper was taken. The soxhlet apparatus was set up and the samples placed in the extractor. One hundred mL of the solvent used was poured into the round bottom flask and the heating mantle was put on while noting the time. The solvent boiled quietly and recycled continuously. This procedure was allowed to run for 2 h for the first run, 4 h for the second runs, 6 h for the third run, 8 h for the fourth run and finally, 10 h for the fifth run. The solvent used and the extracted oil were separated after the experiment. The oil extracted was weighed with the flask and the sample was dried in an oven and weighed again for further analysis (Gunstone and Norris, 1983; Eckey, 1954).

For the determination of saponification value, 1g of the filtered oil samples were poured into 250 mL conical flask. 10 cm³ of 0.5 M alcoholic KOH was added. This was

then fitted with a reflux condenser and the content refluxed by placing the flask in boiling water for half an hour with occasional shaking. The solution was then filtered against the standard 0.5 M HCl using 10 cm³ of phenolphthalein as an indicator. The blank titration was performed in the same way (ISO, 1988a,b, 1975; William, 1966). For the determination of acid value 1g of oil was also dissolved in 250 cm³ of a neutral solvent in a conical flask. The mixture was thoroughly mixed and then titrated with 0.05 M of KOH, 1 mL of phenolphthalein indication solution was used as indicator. The end point reached when a pink colour persisted for 3 sec. The acid number was calculated.

For the determination of iodine value 0.3 g of the oil sample was put in a small glass scoop and inserted into 250 cm³ glass stopped conical flask. 10 cm³ of carbon tetrachloride was dissolved in it. Five mL of Hubl's reagent was added. A stopper was inserted and the content was shaken gently and potassium iodide solution was added. Thirty mL of distilled water was also added. The mixture was titrated with aqueous sodium Thiosulphate solution in the presence of starch solution as an indicator until the blue colour just disappeared. A blank test without the oil was also carried out in the same way. Iodine value was determined. The determination of refractive index was done using refractometer. Some quantity of oil was dropped in the refractometer and the refractive index read (ISO, 1988a,b; 1975).

RESULTS AND DISCUSSION

The results obtained from the analysis performed on the characterization of oil extracted from Acacia Nilotica seeds, based on the choice solvent are given in Table 1-5. Table 1 gives the summary of the result using the three different solvents. In the other tables A, B and C are representative of Petroleum Ether -Ethanol (50:50); Acetone and chloroform methanol, respectively.

Table 1 shows the results of the experiment, the amount of oil extracted from acacia seed for each solvent used and at different times of 2 h intervals. It was noted that the amount of oil extracted increased from the least time of 2 h to the maximum time of 10 h. This shows that the higher the time used the greater the quantity of oil extracted. The reason was that, the more the time used in the experiment, the more surface area of contact between the oil and the solvent

Table 1: Summary of analysis based on solvent used

	Petroleum	Ether-	Chloroform
	ethanol (g)	acetone	methanol
Iodine value	183.525	182.88	187.54
Saponification value	1785.25	1573.61	1983.14
acid value	4.36	5.88	6.44
refractive index	1.33933	1.33563	1.3335

Table 2: Percentage lipid extracted for the three solvents

Time o				ole (W ₁)	Final we	Final weight of sample		% lipid extracted			A 0/15-14
Solvent type	extraction (h)	I	II	IΠ	I	П	Ш	I	II	Ш	Ave. % lipid extracted
Pet. Ether	2	5.84	5.84	5.84	5.74	5.73	5.75	1.71	1.88	1.54	1.71
and ethanol	4	5.84	5.84	5.84	5.63	5.62	5.60	3.60	3.77	4.11	3.83
50:50	6	5.84	5.84	5.84	5.49	5.51	5.50	5.99	5.65	5.82	5.82
	8	5.84	5.84	5.84	5.45	5.42	5.43	6.68	7.53	7.02	7.08
	10	5.84	5.84	5.84	5.42	5.39	5.39	7.19	7.02	7.71	7.31
	2	5.84	5.84	5.84	5.53	5.51	5.54	5.31	5.65	5.14	5.37
	4	5.84	5.84	5.84	5.45	5.42	5.43	6.68	7.19	7.02	6.96
Acetone	6	5.84	5.84	5.84	5.38	5.39	5.40	7.88	7.71	753	7.71
	8	5.84	5.84	5.84	5.35	5.34	5.35	8.39	8.56	8.39	8.45
	10	5.84	5.84	5.84	5.32	5.32	5.33	8.90	8.90	8.73	8.24
Chloroform	2	5.84	5.84	5.84	5.76	5.75	5.77	1.37	1.54	1.20	1.37
and	4	5.84	5.84	5.84	5.60	5.62	5.63	4.11	3.77	3.60	3.83
methanol	6	5.84	5.84	5.84	5.42	5.39	5.40	7.19	7.71	7.53	6.58
50:50	8	5.84	5.84	5.84	5.39	5.36	5.38	7.19	8.22	7.88	7.94
	10	5.84	5.84	5.84	5.36	5.34	5.36	8.22	8.56	8.22	8.33

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Sample	Qty of oil used	Volume of 0.05MKOH used (cm ³)	Blank (cm³)	Acid number	Average acid number
A	1g	2.20	0.20	4.36 mg	5.56
В	1g	2.10	0.20	5.88 mg	
C	1g	2.30	0.20	6.44 mg	

Table 4: Saponification value

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	Qty of oil	Volume of 0.5 HCl ₄	Volume of 0.5m HCl ₄	Saponification	Average saponification
Sample	used (g)	used for oil, V_1 (cm ³)	for the blank, v _o (cm ³)	value (mg)	value (mg)
A	1	75.3	8.80	1785.25	1780.66
В	1	65	8.80	1573.61	
C	1	79.6	8.80	1983.14	

Table 5: Iodine value

	Qty of oil	Volume of sodium	Volume used for		
Sample	used (g)	thiosulphate, v ₄ (cm ³)	blank test, v ₅ (cm ³)	Iodine value (g)	Average iodine (g)
A	0.3	50.55	6.80	183.52	184.64
В	0.3	50.00	6.80	182.88	
C	0.3	51.10	6.80	187.54	

(Perry and Green, 1997; Coulson and Richardson, 1981; Treybal, 1981). It was noted that chloroform-methanol yielded relatively much oil, initially 1.337% at 2 h and 8.33% at 10 h (Table 3-4). It was followed by acetone which started with a yield of 5.37% at 2 h and yielded 8.24% at 10 h. Petroleum ether-ethanol solvent took the least part of the oil yield with the initial output of 1.71% at 2 h and a final output of 7.31% at 10 h. This shows the strong dependence between oil yield, time taken and solvent type. From the beginning of the experiment to 6th there was much quantity of oil to be extracted. At 8 to 10 h, the quantity of oil in the kernel was drastically reduced due to the extraction process. Based on this of extraction of oil from acacia nilotica seed using the above named solvents the optimum time of extraction is 8 h.

The extracted oil was analyzed to determine some of the physical and chemical characteristics of the oil. Table 3 shows the acid value of the oil yielded from their respective solvents A, B and C. The oil sample obtained using chloroform-methanol was noted as the highest with 6.44 mg, followed by acetone solvent, which gave 5.88 mg and the least value was the sample using petroleum ether -ethanol, with 4.36 mg, the standard for acid value of organic oil is 2 mg. In the saponification value of oil for the 3 samples, the sample of oil using acetone, gave the least value while that using chloroform-methanol gave the highest value. The standard value of saponification for organic oil is between 185 to 196 mg (ISO, 1988a, b, 1975).

Table 5 shows the iodine value for the oil of the 3 samples. Acetone sample gave the least value of 182.88 g and chloroform-methanol sample gave the highest value of 187.54 g. The standard of iodine value for organic oil is between 125 to 150 g. For the refractive index, one of the physical characteristics of the oil was determined for the 3 different samples and was found to be 1.33933 for the sample of petroleum ethe-ethanol; 1.33563 for the sample of acetone and 1.3335 for the sample of chloroform-methanol. The standard value for refractive index of organic oil is 1.3 to 1.6. It was noted that the saponification value, iodine value, and acid value were above the standard values of organic oil. This was due to

the fact that the oil was not refined; the presences of impurities in the oil gave out inappropriate results. The result of the index obtained falls within the standard value which is in the range of 1.3 to 1.6 (ISO, 1988a, b, 1975; Gunstone and Norris, 1983).

CONCLUSION

Petroleum ether-ethanol, acetone and chloroformmethanol were used to extract oil from Acacia Nilotica seeds. Three replicates of sample were used and an average percentage of oil extracted of the replicates were calculated. For 2 h extraction, the amount of oil obtained were 1.71, 5.37 and 1.54%, respectively for the three solvents. For 4 h of extraction the amount of oil obtained were 3.83., 6.96 and 3.83%, respectively. For 6 h extraction, the amount of oil obtained were 5.82, 7.71 and 6.58%, respectively. For 8th extraction, the amount of oil obtained were 7.08, 8.56 and 7.94%, respectively. And finally, for 10th extraction, the amount of oil obtained were 7.31, 8.45 and 8.33%, respectively. The acid values of oil yielded using petroleum ether-ethanol (50:50), Acetone and chloroform-methanol are 4.36 mg, 5.88 mg and 6.44 mg, respectively. Saponification value obtained were 1785.25 mg, 1573.61 mg and 1983.14 mg, respectively. In the same way, iodine values were determined as 183.5 g, 182.88 g and 187.54 g, respectively.

REFERENCES

- Alfred, I.I. and O.N. Patrick, 1985. Integrated Food Science and Technology for the Tropics, International Cole Edition, Macmillan publishers, pp. 78-80.
- Coulson, J.M. and J.F. Richardson, 1981. Particle Technology and Separation Processes, chemical Engineering, (4th Edn.), Butterworth-Heinemann Pub., Oxford.
- Eckey, E., 1954. Vegetable Fats and Oils, American Chemical Society Monograph No. 123, USA.
- Femando, A. and E.O. Akuyobi, 1987. Chemical Analysis of Vegetable Oils in Sokoto State, Nigeria, J. Basic and Applied Sci. Nigeria, pp: 11-14.

- Gavriel, S., 1996. Handbook of Industrial Engineering, John Wiley and Sons, New York, pp: 132.11.1-13.11.15.
- Gunstone, F.D. and F.A. Norris, 1983. Lipids in Foods Chemistry, Biochemistry and Technology, (1st Edn.), Pergamon Publisher, New York, pp. 108-121.
- International Standard Organisation, I.S.O., 1975. Animal and Vegetable Fats and Oils, Determination of Peroxide Values, ISO 3960 bulletin, (1st Edn.), pp. 1-3.
- International Standard Organisation, I.S.O., 1988b.

 Animal and Vegetable Fats and Oils, Determination of Saponification Value, ISO 3657 bulletin, (2nd Edn.), pp. 1-2.
- International Standard Organization, I.S.O., 1988a.

 Determination of the Moisture and Volatile Matter
 Content of Animal, Vegetable, Oils and Fats, ISO 662
 bulletin, (2nd Edn.), pp: 1-2.
- Macreie, R., R.K. Robinson and M.J. Sadler, 1993.
 Encyclopaedia of Food Science, Food Tech. Nutr.
 Acad. Press Ltd, N.Y., pp. 26-27.
- Paterson, H.B.W., 1989. Handling and storage of oil seeds, Oils, Fts and Meal, Elsevier Applied Sciences, London.
- Perry, R.H. and D.W. Green, 1997. Perry Chemical Engineers' Handbook, (7th Edn.), McGraw Hill Companies, pp. 2.53,2.33,2.207.
- Scheitzer, P.A., 1988. Handbook of Separation Techniques for Chemical Engineers, (2nd Edn.), James Peter Associate Inc, McGraw hill Inc., USA, pp. 1-217.
- Treybal, R.E., 1981. Mass Transfer Operation, (3rd Edn.), Departmental Student Edition, Mc Graw-Hill, Tokyo.
- Weiss, E.A., 2000. Oil seed Crops (2nd Edn.), World Agricultural Series, Black Well Science Publication, United Kingdom, pp. 287-329.
- William, K.A., 1966. Oils, Fats and Fatty Foods (Their Practical Application), (4th Edn.), Churchill Pub. U.K., 2: 61-65.