Solvent Extraction of the Oils of Rubber, Melon, Pumpkin and Oilbean Seeds

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Solvents of differing dielectric constant were used to extract oils from the seeds of: rubber [*Hevea brasiliensis* (Kunth) Muell. Arg.], melon [*Colocynthis vulgaris* Schrad], fluted pumpkin [*Telfairia occidentalis* Hook f.] and oilbean [*Pentaclethra macrophylla* Benth]. The aim was to examine the effect of solvent polarity on oil yield and oil properties.

The oils were extracted under Soxhlet conditions with the following solvents: petroleum benzene (60 – 80°C), cyclohexane, isopropyl ether, ethyl acetate, tetrahydrofuran, propan-2-ol and acetone. The oils were characterized by acid number, iodine value and color intensity determinations.

The oil yields of each seed in different solvents ranged as follows: 58.0–64.4% (pumpkin), 56.1–59.1% (melon), 40.6–48.8% (rubber) and 35.4–43.3% (oilbean). The equilibrium extracting capacity of each solvent was found to depend on two factors, namely, the nature of the oil and the polarity of the solvent. Both factors were found to determine the acid number, iodine value and color intensity of each oil.

Two methods are generally used for extraction of oil from oil-bearing seeds, namely, solvent extraction and mechanical expression. Solvent extraction has the advantage of giving higher oil yields than mechanical expression. However, the use of solvents for oil seed extraction has been criticized because of toxicity and problems associated with solvent flammability and recovery (1). The press method for oil extraction has been considered more economical and capable of yielding better values for oil constants. It has the additional advantage of requiring considerably less equipment, space and time (2). However, solvent-extracted oils of comparable qualities to those obtained by expression are now obtainable, and new, relatively safe solvents, such as hexane and ethylene chloride (3) are also available. In industry, a combination of both mechanical expression and solvent extraction is the most commonly used method.

A number of factors determine the choice of solvent for seed oil extraction, notably, solvent extraction capacity, effects of solvent on oil properties, process safety, solvent volatility and stability, and economic considerations. This work intends to focus on the first two parameters. Solvent extraction capacity and solvent effect on oil properties have received attention in the literature (4,5). The extractability of an oil has been found to depend on the nature of the oil, the nature of the solvent, the temperature and time of contact between the solvent and the feed, flake thickness and pretreatment conditions of the seed. Owing to the differences in solvent capacity, the non-fatty materials extracted along with the oil vary, leading to differences in the quality of the oil extracted. It is the objective of this work to provide information on solvent capacity and solvent effects in the extraction of four native oils that have good commercial potential, as edible oils or as drying or semidrying oils in the coatings industry. These are the oils of rubber seed [*Hevea brasiliensis* (Kunth) Muell. Arg.], pumpkin seed [*Telfairia occidentalis* Hook f.], melon seed [*Colocynthis vulgaris* Schrad], and oil bean [*Pentaclethra macrophylla* Benth].

Specifically, the work attempts to establish the level of oil yield obtainable from each oil-bearing seed by using solvents of different boiling points and polarity; to correlate the oil yield with some solvent property such as dielectric constant with a view to understanding solvent/oil interaction; and to establish the effects of extracting solvent on such oil properties as percentage free fatty acid, degree of unsaturation and level of coloring matter.

MATERIALS

Seed samples. Rubber seeds were obtained from the Nigerian Rubber Board (Benin City, Nigeria). The other three seeds were purchased from a local market. The seeds were shelled, dried in an oven at 60°C to constant weight and were then ground for extraction. The moisture contents of the dried seeds before grinding were rubber (5.1%), melon (4.9%), pumpkin (5.7%) and oilbean (6.6%).

Extraction solvents. The following analytical grade solvents were used as purchased for the extraction: petroleum benzene (60–80°C), cyclohexane, isopropyl ether, ethyl acetate, tetrahydrofuran, propan-2-ol and acetone. The solvents were selected mainly on the basis of their dielectric constants.

Oil extraction procedure. The extraction of oil from the above oil-bearing seeds was carried out with a Soxhlet extractor. 100 g of the shelled and ground seed meal was packed into a weighed thimble, which was then introduced into the extractor. The extraction was carried out for six hours for each solvent/oil seed system. The temperature of extraction corresponded with the boiling point of the solvent in use. Solutions of the extracted oils were stripped of their solvents by vacuum distillation at a reduced temperature. The oil obtained was weighed, and the percentage oil yield calculated. The extraction of the oil of each seed with each solvent was done in three separate batches to obtain an average oil yield.

Characterization of the oils (acid number). This was determined according to ASTM method (6).

Iodine value. The iodine value of each oil sample was determined by the WIJS method (7).

Color intensity of oil. The level of coloring matter in each oil was determined by absorbance measurement in a photoelectric colorimeter (Model AE 11), using a 420nm filter. Water was used as the reference liquid.

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RESULTS AND DISCUSSION

Oil yield and its correlation with solvent dielectric constant. The oil yields obtained under Soxhlet conditions for each of the oil-bearing seeds of rubber, pumpkin, melon and oilbean in different solvents are given in Table 1. The figures are based on the mass of shelled dry seed. The error figures calculated as standard deviations for the four oils are given against the oil yields. The results show that the oil contents of the seeds are in the order: pumpkin > melon > rubber > oilbean.

The oil extraction performance of each solvent appears to be generally dependent on the nature of the oil because solvent extraction capacity varies from oil to oil. For instance, while ethyl acetate and tetrahydrofuran gave the highest oil yields in rubber, their oil yields in melon gave next to lowest value. This oil effect may be due to the differences in the drying nature of the oils, which is related to the varying levels of unsaturated fatty acid chains as shown in Table 2.

The oil yields of a given seed, however, show modest differences as the solvent is varied, which can be attributed to some solvent property. Consequently, plots of oil yields versus solvent bulk dielectric constant have been carried out in Figure 1 in order to examine whether solvent polarity is the property responsible for the observed differences. Considering the error figures in Table 1, the plots seem to show that, in general, solvents of dielectric constant of between 6 and 8 give higher oil yields than those of either lower or higher dielectric constants. This may be attributed to the amphipathic nature of the triglycerides which have both polar and nonpolar components. In nonpolar solvents (low dielectric constant) solubility will be impaired by the polar ester component of the trigylceride, while in polar solvents solubility will be adversely affected by the nonpolar groups (the fatty acid chains) of the triglycerides. Thus, both cyclohexane and acetone in general exhibit lower oil yields than solvents of intermediate polarity. The high oil yields obtained with propan-2-ol, despite its high bulk dielectric constant, may be due to some specific interaction involving hydrogen bonding with the ester groups of the triglycerides.

Solvent effect on oil acid value. The acid value of an oil depends largely on the age and species of the seed from which the oil is extracted. However, the interest here is to examine any possible solvent effects, specific or bulk, on the level of free fatty acid extracted from a given oil of a particular age and species. The results of acid number determinations for each oil extracted with different solvents are given in Table 3, together with the standard deviations. An examination of the acid values reveals that the level of free fatty acid (ffa) extractable by a given solvent is dependent on the nature of the oil. For instance, while cyclohexane gave the highest ffa from rubber seed, it resulted in the lowest from melon seed; similarly, while THF extracted the highest ffa in pumpkin, it gave the least in rubber.

Plots of acid values versus dielectric constants are given in Figure 2 in order to reveal any solvent effect.

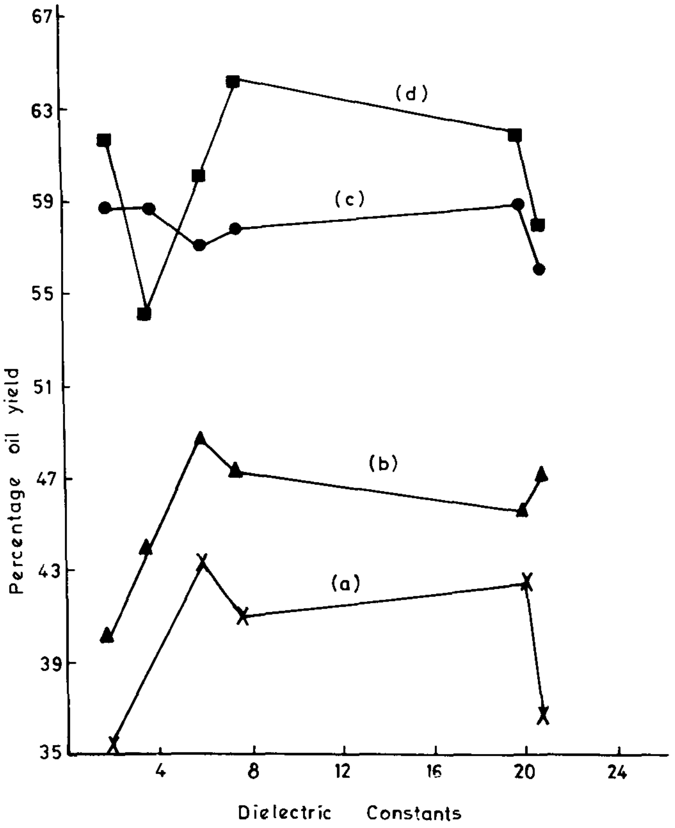

Fig. 1. Plots of percent oil yield vs solvent dielectric constant: (a) oilbean seed, (b) rubber seed, (c) melon seed, and (d) pumpkin seed.

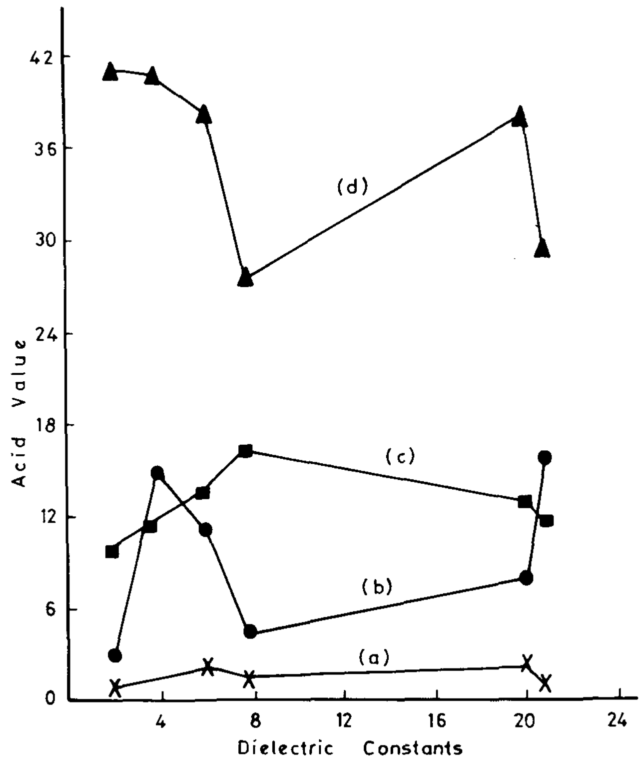

Fig. 2. Plots of oil acid value vs solvent dielectric constant: (a) oilbean seed, (b) melon seed, (c) pumpkin seed, and (d) rubber seed.

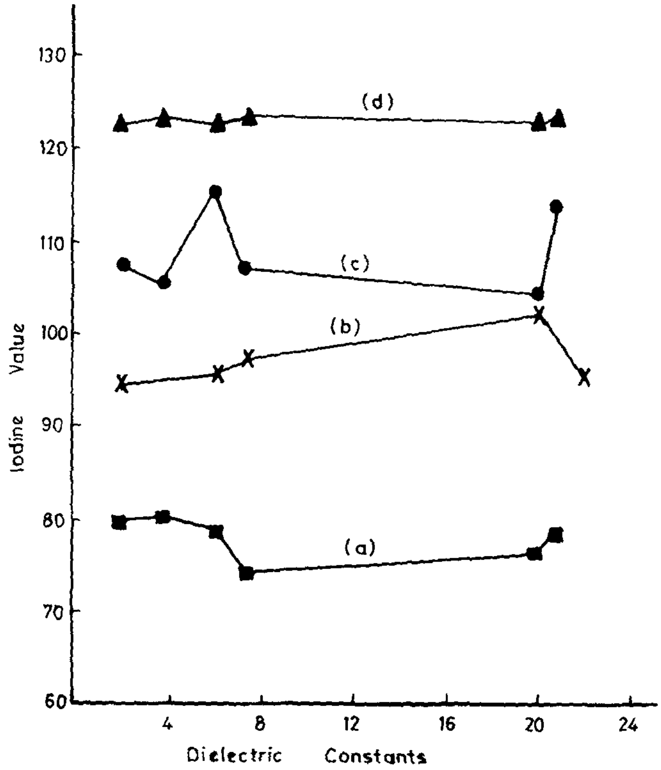

Fig. 3. Plots of oil iodine value vs solvent dielectric constant: (a) pumpkin seed, (b) oilbean seed, (c) melon seed, and (d) rubber seed.

With the exception of rubber seed, the hydrocarbon solvents appear to extract less free fatty acid than the polar solvents. The Figure shows an initial rise in acid number with increase in dielectric constant (ϵ), with some maximum in the region of between 4 and 6. Again, the high ffa values obtained with propan-2-ol may be due to specific interaction. On the whole, however, there seems to be no clear-cut dielectric effect on ffa.

Solvent effect on oil iodine value. Iodine value represents the degree of unsaturation of the oil and the free fatty acids. The results of the iodine value determinations and standard deviations are given in Table 4 for the four oils using different solvents. Plots of the iodine values versus the dielectric constant of the solvent are shown in Figure 3. With the exception of melon seed oil extracts of ethyl acetate and acetone and oilbean seed oil extract of propan-2-ol, the iodine values of the oils seem to show no significant changes with dielectric constant of the extracting solvents. The slight variations in iodine value observed in the above-mentioned exceptions may arise from the extraction of varying amounts of unsaturated free fatty acids. Also, increased levels of coloring matter, some of which contain unsaturated bonds, could lead to increase in iodine value.

Solvent effect on oil color intensity. The coloring matter content, and hence, the color intensity of the oils, were determined by UV absorbance measurements, and the results are given in Table 5. As before, oil effect appears evident as most solvents change position in their ability to extract coloring matter as we go from one oil to another. Solvent effect is also apparent with propan-2-ol, ethyl acetate, THF and acetone showing greater tendency to remove coloring matter than the other solvents.

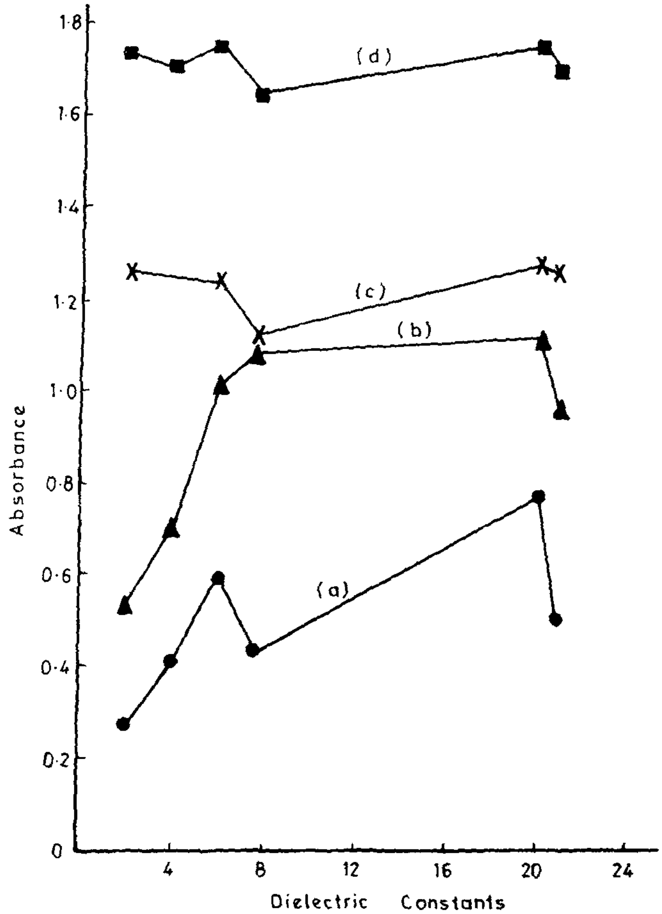

Fig. 4. Plots of oil absorbance value vs solvent dielectric constant: (a) melon seed, (b) rubber seed, (c) oilbean seed, and (d) pumpkin seed.

The effect of dielectric constant on the degree of coloring matter extracted is shown in Figure 4. For oils of rubber and melon seeds, coloring matter content seems to increase generally with an increase in dielectric constant, with the usual deviations arising from possible specific interaction effects as typified by the behavior of propan-2-ol.

REFERENCES

1. Brats, G., *Rev. Fr. Corps Gras. 25*:3 (1978).
2. Nur, Idris Mohammed, *J. Assoc. Adv. Agric. Sci. Afr. 3*:46 (1976).
3. Koller, G., Seventh Int. Cong. Appl. Chem. London. Abst. in *J. Soc. Chem. Ind., 28*:19 (1909).
4. Arnold, L.K., *Proc. Iowa Acad. Sci. 51*:309 (1944).
5. Sievens, A.F. and T.D. McIntyre, *Cotton Oil Press, 4*:44 (1921).
6. *Annual ASTM Standards-Paint Tests for Formulated Products and Applied Coatings*, Part 21 (1973).
7. Lewkowitsch, J.I., *Chemical Technology and Analysis of Oils, Fats and Waxes*, 5th ed., Macmillan, London, 1973.
8. Eka, O.U., *West Afri. Biol. Appl. Chem. 20*:45 (1977).
9. Girgis, P., and F. Said, *J. Sci. Food Agric. 19*:615 (1968).
10. Coursey, D.G., *A Review of the Lesser Known Vegetable Oils and Oil Seeds Available in Nigeria*, Federal Institute of Industrial Research, Oshodi, Nigeria, Technical Memorandum, No. 22, 1964.
11. Asiegbu, J.E., *J. Sci. Food Agric. 40*:151 (1987).

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