

A COMPARATIVE STUDY OF THE EXTRACTION AND CHARACTERISATION OF OILS FROM *Glycine max. L* (SOYA BEAN SEED), *Elaeis guineensis* (PALM KERNEL SEED) AND *Cocos nucifera* (COCONUT)

ABSTRACT

Aims: To extract and characterize oils from *glycine max. l* (soya bean seed), *elaeis guineensis* (palm kernel seed) and *cocos nucifera* (coconut) using ethanol and n-hexane.

Place and duration of study: Department Industrial Chemistry, Renaissance University, Ugbawka, between April and July 2018.

Methodology: 100g of the milled seed sample (soya bean, coconut and palm kernel) were bagged and used for the extraction process; using a Soxhlet apparatus. 150ml of each solvent (n-Hexane and Ethanol) was measured into 250ml flat bottom flask and heated at a constant temperature of 70°C using n-Hexane and 80°C using Ethanol to reflux. Physicochemical analysis which includes: Acid values, iodine value, free fatty acid (% oleic) and saponification value was carried out on the extracted oil.

Results: The percentage oil yield from ethanol was 8.58%, 26.01% and 31.21% for soya bean, coconut and palm kernel respectively while that of n-Hexane was 14.69%, 31.85% and 45.28%. The seed oils extracted using n-Hexane had a clear yellowish colour, while those extracted with ethanol had a dark brown colour. Free fatty acid values were the same in n-Hexane and ethanol extracts of soya bean oil. These values were slightly higher in the coconut and palm kernel oil extracts using ethanol. Acid values were observed to be slightly higher in the ethanol extracted oils. The Saponification values were generally higher in ethanol extracted oils than in hexane extracted oils. There was maximum oil recovered with hexane than ethanol when the extraction process was carried out for 120 minutes.

Conclusion: In spite of the fact that n-hexane gave a better extraction yield, ethanol appeared to be a better alternative for the extraction process because it is green, less toxic and safer to handle.

Keywords: Extraction; Characterization; Solvent; Soxhlet; n-Hexane; Ethanol

1. INTRODUCTION

There has been an increase in the world production of oilseeds over the last thirty years [16], this would appear to be related to the increasing demand for oilseed products and by-products as oilseeds are primarily grown for their oil and meal [4]. Oils from most edible oilseeds are used in the food industry, though there is growing emphasis on industrial utilization as feedstock for several industries with about 80% of the world production of vegetable oils for human consumption. The remaining 20% utilization is between animal and chemical industries [10]. According to Takads [13], bio-oils from oilseeds are used as Straight Vegetable Oil (SVO) or as biodiesel (trans esterified oil) depending on type of engine and level of blend of the oil; soya bean oil is not an exception. This phenomenon has created a school of thought that it is better to use oilseeds as bio-fuel, which will lessen the competition for fossil fuels, which are not renewable [6]. Fossil fuels are not only costly in terms of price but are also costly to the environment as they degrade land, pollute water and cause a general destabilization of the ecosystem with global

warming as an end result [2]. Furthermore, crude oil yields socio-political power that often dictates the pace of economic growth in specific locations, especially non-oil producing nations. Nevertheless, the petroleum industry requires a greater quantity of oil to meet its demand. In order to meet the required amounts needed by all industries, these fats and oils must be available in large quantities locally with an effective extraction process at an affordable cost [15].

The ability of a particular oilseed to fit into the growing industries depends on its utilisation potential, rate of production, availability and ease of the processing technology. Thus while some oilseeds are being largely utilised in the oil processing industries, quite a number of oilseeds are under-exploited. Soya bean (*Glycine max. L*), coconut (*Cocos nucifera*) and palm kernel (*Elaeis guineensis*) are important oilseed crops in the world, they are rich in protein and oil 14-20% in soya bean, about 45% in palm kernel, and 30% in coconut [15]. Oil can be extracted from these seeds by using traditional methods of extraction (on a very small scale), mechanical expression (hydraulic and screw presses) which can be manual, semi-automated or automated, and solvent extraction (e.g. hexane, fluid carbon dioxide) or a combination of two of these methods. The efficiency of traditional methods of extracting the oil is low (less than 50%), labour intensive, time consuming and possibly compromising quality and safety standards [5]. Mechanical pressing of oilseeds is the most common method of edible oil extraction used in the world [7]. The main reason for this is that it provides a non-contaminated, protein-rich low-fat deoiled cake, an important byproduct in many developing nations at a relatively low cost. However, mechanical presses do not have high extraction efficiencies, even when using hydrothermally treated oilseeds. Extraction efficiencies seldom exceed 80%, compared to over 98% achieved by the solvent extraction method [3]. Solvent extraction is capable of removing virtually all of the oil available in the cells of the oilseed though the equipment required is generally too expensive to acquire and there is the inherent danger of fire explosion due to the chemicals used in the extraction process [12]. The process of solvent extraction, however, is faster and less expensive compared to mechanical extraction. Conventionally, soya beans are subjected to solvent extraction because of the low oil content (about 18-20%) and relatively lower oil recoveries in mechanical expression systems. Solvent extraction, in general, implies the removal of miscellanea from an insoluble solid phase by dissolution in a liquid solvent [13]. Literature on the exact quantities of oil extracted per seed conditioning and kind of solvent apart from hexane is unknown. Hexane however, is classified as being hazardous to working staff and based on this assertion has been banned by the government of the United States of America. It is therefore important to determine the extraction rates for other suitable solvents such as ethanol, which are available at relatively cheaper prices [4].

Increasing the extraction of oil to meet the rising demand for vegetable oils in different industries requires a suitable solvent which is readily available in the country at a relatively cheaper cost [6]. This study therefore, sought to provide a fair idea on a possible solvent replacement using ethanol which is available, has ability to extract oil from vegetable seeds and also affordable over hexane, which is considered hazardous, expensive and occasionally scarce based on demand and cost of petroleum, in the extraction of palm kernel, coconut and soya bean oils giving a clear indication as to the best options and requirements for higher optimum oil recoveries from these seeds using these two solvents [11].

2. MATERIAL AND METHODS

2.1 Sample Collection and Preparation

The samples used in this study- oil bean seed, palm kernel seed and coconut were collect from Ogbete Main Market Enugu, Nigeria. The samples were washed with distilled water and oven dried at a temperature of 80°C for 5hrs. The samples were further milled until fine particle size was achieved. 100g of the milled seed sample (soya bean, coconut and palm kernel) were bagged and used for the extraction process.

2.2 Solvent Extraction Process

Extraction was done, using a Soxhlet apparatus. 150ml of each solvent (n-Hexane and Ethanol) was measured into 250ml flat bottom flask and heated at a constant temperature of 70 °C using n-Hexane and 80 °C using Ethanol to reflux. The heat caused the solvent to vapourise through the thimble containing the sample as the solvent boiled in the flask; the vapour was trapped and cooled by the condenser above the thimble. The cooling turned the vapour into warm liquid which hydrolyzed the sample in the thimble. When the thimble was filled with the drops of the warm solvent from the condenser, the solvent (which contained traces of the oil) was poured out into the flat bottom flask beneath the thimble containing either

n-Hexane or Ethanol automatically through a siphon arm. The process was continued for the duration of 2hrs for each sample. At the end of each extraction process, the milled sample was removed from the thimble and the extraction process repeated, but this time for a different sample seed. The oil was poured into a weighed empty beaker and placed in a steam bath at a controlled temperature of 65°C. At the end of each extraction, the weight of the extract was multiplied by 100 and divided by the original mass of the sample (100g) to give the oil yield.

2.3 Determination of Oil Quality

Several indices are used for the determination of oil quality. For the purpose of this experiment, free fatty acid, iodine value, acid value and saponification value were determined.

2.3.1 Free Fatty Acid

Free fatty acid is the percentage by weight of a specified fatty acid (e.g. % oleic acid) [7]. High levels of free fatty acids especially linoleic acids are undesirable in finished oils because they can cause off-favours and shorten the shelf life of oils. The free fatty acid was determined according to the Official Method Ca 5a-40 of American Oil Chemists' Society AOCs (1993) [1]. About 0.5 g of the extracted oil sample was weighed into a 250 ml Erlenmeyer flask using an analytical balance. 20 ml of 95% neutralized ethanol was added to the flask. The solution was heated slightly at 20°C to aid the dissolution of the fat in the alcohol. 3 drops of phenolphthalein solution were added as indicator. The obtained yellowish solution was titrated with 0.1M standard sodium hydroxide solution while shaking the solution vigorously. The colour of the solution turned pink and at the point when the pink colour persisted for 30s was termed the end point. The percentage of free fatty acid in the oil was calculated as follows:

$$\% \text{ Free Fatty Acid} = \frac{T.V \times M \times 28.2}{\text{Weight of sample}}$$

where,

T.V = average volume of NaOH (ml),
M = Molarity of NaOH (0.1),

2.3.2 Iodine Value

Iodine value is a measure of the unsaturation of fats and oils, and is expressed in terms of the number of centigrammes of iodine absorbed per gramme of the sample (% iodine absorbed) during oxidation, which consumes the double bonds resulting in a reduction in iodine. It is an indicator for double bindings in the molecular structure, which influences the long term stability properties of the oil (important for storage). Oils having high iodine number are polyunsaturated indicating the degree of unsaturation and are desired by oil processors, while a lower iodine number is indicative of lower quality [8]. The iodine value was determined according to Official Method Cd 1-25 of AOCs (1993) [1]. 0.5 g of the filtered oil sample was weighed into a 500 ml flask. 15ml of tetrachloride was added to the sample and swirled to ensure that the sample was completely dissolved in the tetrachloride. 25 mL of Wijs solution was dispensed into the flask containing the sample and swirled to ensure an intimate mixture. The flask with content was immediately kept in a dark place at a temperature of about 25°C – 30°C for 30 mins. The flask was removed and 20 mL of 10% potassium iodide (KI) solution added followed by an addition of 150 mL of distilled water. The solution was titrated with 0.1M Sodium thiosulphate (Na₂S₂O₃) solution while shaking it constantly and vigorously until the reddish colour had almost disappeared. 5 mL of 1% starch indicator was added and the titration continued until the blue-black colour just disappeared. A blank was preformed alongside.

$$\text{Iodine Value} = \frac{(T.V_2 - T.V_1) \times M \times 12.69}{\text{Weight of sample used}}$$

where,

T.V₂ = Titration of Na₂S₂O₃ blank (ml),
T.V₁ = Titration of Na₂S₂O₃ sample (ml),
M = Molarity of Na₂S₂O₃ solution (ml),

2.3.3 Acid Value

Acid value is the number of milligrammes of potassium hydroxide necessary to neutralize the free acids in one gram of oil sample. The samples that contain virtually no free acids other than fatty acids, the acid value may be directly converted by means of a suitable factor to percent free fatty acids. Where vegetable

oils are used as lubrication products, the acid value can affect the properties of the lubrication oil, if larger quantities reach the oil sump.

The Acid value was determined according to the Official Method Ca 5a-40 of American Oil Chemists' Society (AOCS) (1993) [1]. About 0.5 g of the extracted oil sample was weighed into a 250 ml Erlenmeyer flask using an analytical balance. 20 ml of 95% neutralized ethanol was added to the flask. The solution was heated slightly at 20°C to aid the dissolution of the fat in the alcohol. 3 drops of phenolphthalein solution were added as indicator. The obtained yellowish solution was titrated with 0.1M standard sodium hydroxide solution while shaking the solution vigorously. The colour of the solution turned pink and at the point when the pink colour persisted for 30s was termed the end point. The percentage of Acid in the oil was calculated as follows:

$$\text{Acid Value} = \frac{(A - B) \times (M) \times 56.1}{\text{Weight of sample used}}$$

Where,

A = NaOH used in titration (ml)

B = NaOH used in the blank (ml)

M = Molarity of NaOH

2.3.4 Saponification Value

Saponification is the chemical reaction in which an ester is heated with an alkali (especially the alkaline hydrolysis of a fat or oil to make soap). Saponification value is however, the amount of alkali necessary to saponify a definite quantity of oil sample. In combination with acid values, saponification values are useful in providing information as to the quantity, type of glycerides and mean weight of the acids in a given sample of oil. Saponification is only of interest if the oil is for industrial purposes, as it has no nutritional significance. But due to the fact that each fat has within the limits of biological variation, a constant fatty acid composition determination of the saponification value is a reasonable means of characterizing the fat [2].

The saponification value was determined using Official Method Cd 3- 25 AOCS (1993) [1]. 0.5 g of the dried and filtered oil sample was weighed into a 250 ml Erlenmeyer flask. 50mls of 0.5M ethanolic KOH was added. The content was refluxed and allowed to stand for 15 mins. A blank was conducted simultaneously where all reagents were added with the exception of the oil sample. 1 ml of phenolphthalein indicator was added to the sample and the sample titrated with 0.5M hydrogen chloride (HCl) until the pink colour just disappeared and the volume of the HCl recorded.

$$\text{Saponification Value} = \frac{(T. V_2 - T. V_1) \times M \times 65.1}{\text{Weight of sample used}}$$

where,

T.V₂ = 0.5M HCl required to titrate blank (ml),

T.V₁ = 0.5M HCl required to titrate sample (ml),

M = Molarity of HCl solution (ml)

3. RESULTS AND DISCUSSION

3.1 OIL EXTRACTION YIELD

Comparing oil extracts in Fig.1, there were significant differences between mean yields from the solvents n-Hexane and Ethanol. With an average extraction of 14.69%, 31.85% and 45.28% for soya bean, coconut and palm kernel respectively using n-Hexane and 8.58%, 26.01% and 31.21% for soya bean, coconut and palm kernel respectively using ethanol, n-Hexane yielded relatively higher oil compared with ethanol. Palm kernel yielded the highest amount of oil closely followed by coconut, and soya bean having the lowest oil yield.

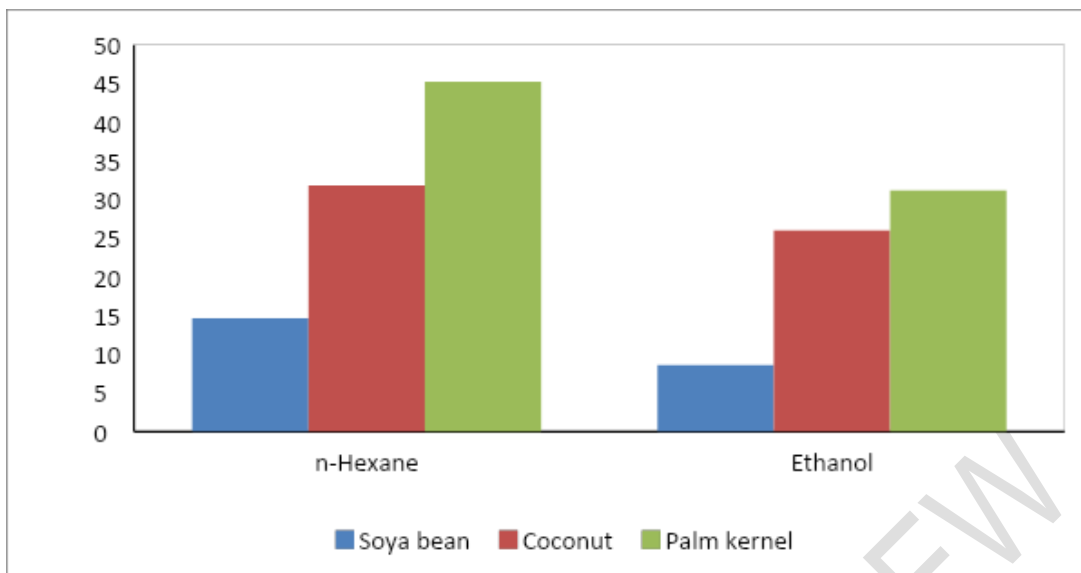


Fig.1. Comparative chart of Oil extraction yields of the seeds per solvent

3.2 Free Fatty Acid Value

Free fatty acid is the percentage by weight of a specified fatty acid. High concentrations of free fatty acids are undesirable in crude oils because they result in large losses of the neutral oil during refining. Free fatty acid values were the same in n-Hexane and ethanol extracts of soya bean oil. These values were slightly higher in the coconut and palm kernel oil extracts using ethanol. The oils might contain some impurities since they are in a crude form, which through the help of the ethanol could cause the hydrolysis of the ester linkage thereby increasing the free fatty acid level. However, the free fatty acid values obtained from this work were low implying low rancidity of the oil and thus viable as edible oil.

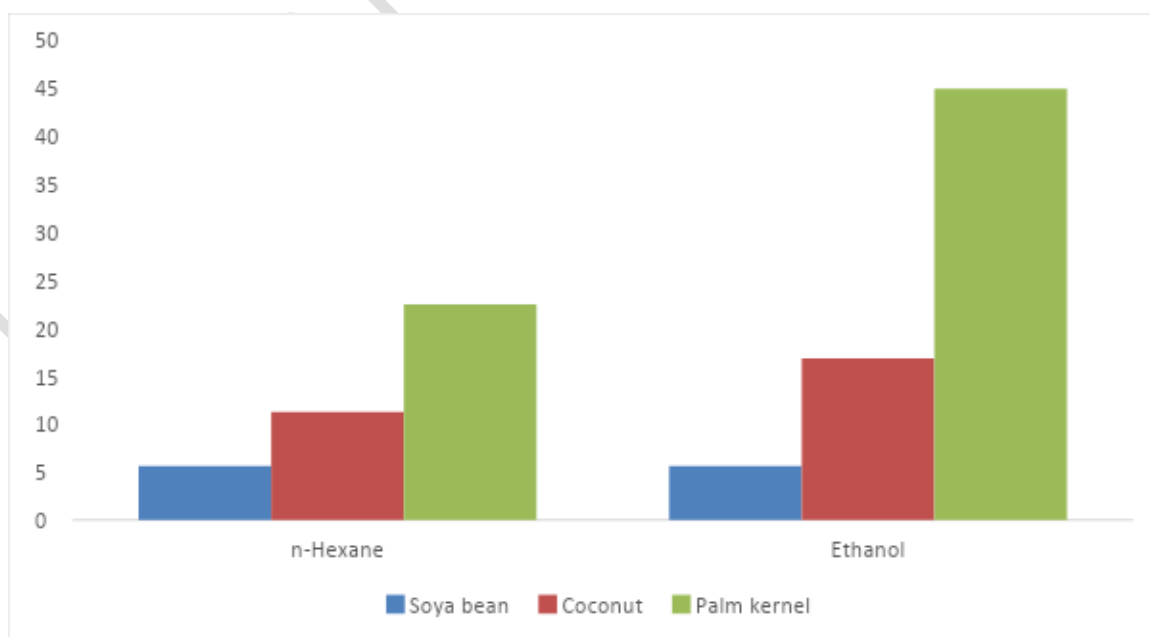


Fig. 2: Comparative Chart of the Free Fatty Acid Value of the Oils

3.3 Acid Value

Acid values are observed to be slightly higher in the ethanol extracted oils. This could be due to the hydrolysis of the oils through the solvent. But the overall low acid value of the oils implies a good stability of the oils.

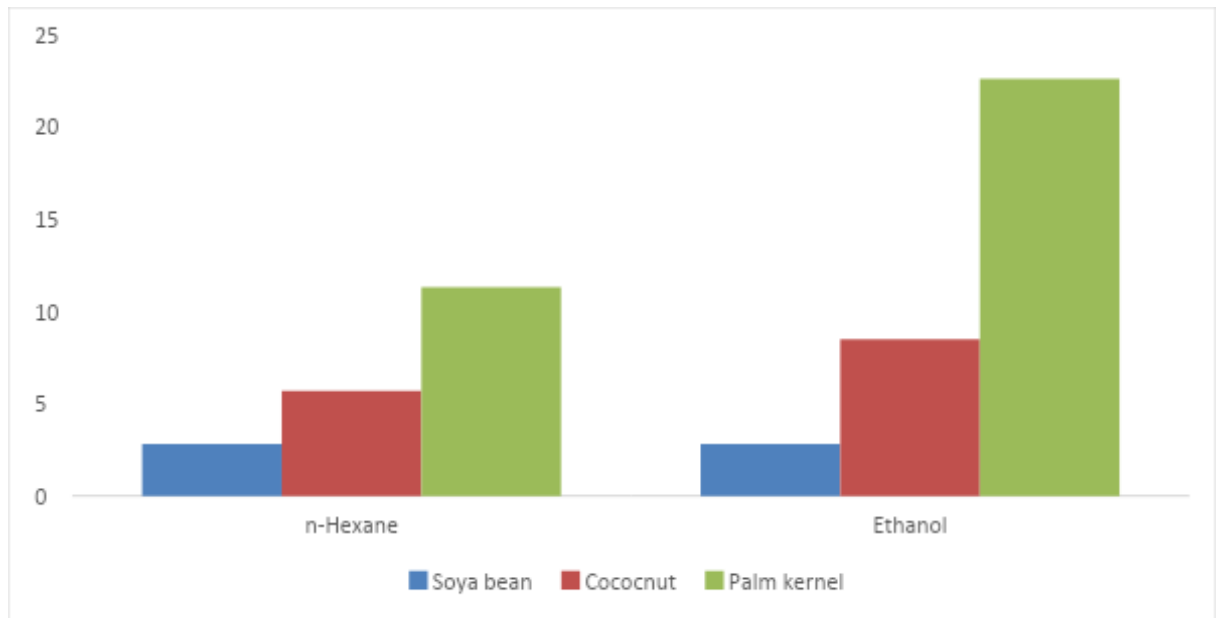


Fig. 3: Comparative Chart of Acid Value of the Oils

3.4 Saponification Value

The saponification value gives information concerning the character of the fatty acid of the fat: the longer the longer the carbon chain, the smaller the saponification value. These values are generally higher in ethanol extracted oils



Fig.4: Comparative Chart of the Saponification Value of the Oils

3.5 Iodine Value

The results of the iodine value were less than 100mg iodine/100g meaning they have very few double bonds and are therefore saturated. They are then classified as non-saturated oils and can be recommended for people with dry skin.

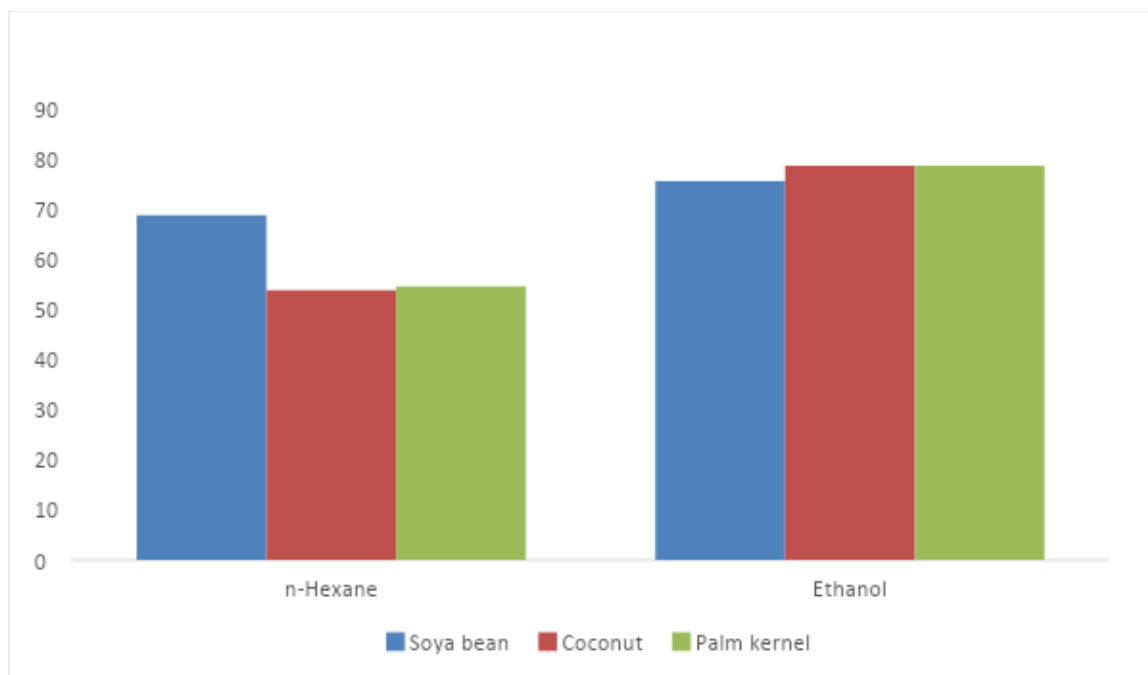


Fig. 5: Comparative chart of the Iodine Value of the Oils

3.2. Oil yields

For maximum oil yield, important parameters were taken into consideration:

3.2.1 Effect of extraction time

The extraction time is an important parameter for solvent extraction. It helps in deciding the optimum residence time required in extractor. It is observed that extraction of oil increased with time for both the solvents. It is also observed that the rate of extraction is very high during the first 20 minutes of extraction and afterwards it tapers off. There is maximum oil recovered with hexane than ethanol when the extraction process was carried out for 120 minutes. 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 the fresh solvent. The later slower rate may be attributed to lower driving force due to increasing oil concentration in the solvent [14].

3.2.2 Effect of particle size

The extraction was carried out from the milled seeds of the palm kernel, coconut and soya bean. Lower extraction rate can be attributed to the fact that the bigger particles have less surface area directly exposed to the solvent as compared to the smaller size and the solvent has to penetrate into the core of the seed to leach the oil out of the seed which restricts the rate of oil extraction from the bigger seed particles. Thus the seeds used for this work had to be milled to very fine powdered particles for maximum yield of the oils.

3.2.3 Colour

The colour of the oils extracted with ethanol was dark while the that of oils extracted with n-Hexane was lighter.

3.2.4 Odour

The odour of the coconut oil was that of coconut smell, the palm kernel oil was that of palm kernel smell and the soya bean oil had relatively no smell. This result is due to the fact that the oils were not refined, bleached or deodorized.

4. CONCLUSION

The result of the solvent extraction of oils from Soya bean (*Glycine max. L*), coconut (*Cocos nucifera*) and palm kernel (*Elaeis guineensis*) oilseeds using ethanol and n-hexane, showed that although n-Hexane has a better extraction yield, ethanol, a green and safe solvent can be a better alternative to n-hexane due to its low toxicity and the fact that it is less hazardous for the environment.

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