Global Scientific JOURNALS
GSJ: Volume 9, Issue 11, November 2021, Online: ISSN 2320-9186
www.globalscientificjournal.com

Qualitative Analysis Extract Recovery from Melon Seed at Different Temperatures Using Hexane and Ethanol as Solvents

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ABSTRACT

Solvent Extraction and Characterization of Vegetable Oil from CUCUMEROPSIS MANNII was considered in this work. Hexane and Ethanol were used as solvent for the extraction process and the desired vegetable oil product obtained from steam distillation process of melon seed through the application of Soxhlet Extractor apparatus. The oil obtained from the process was characterised for its suitability for human consumption and compared with commercially available vegetable oil in the market. This research work revealed the order of magnitude of conventional, Hexane and Ethanol as solvent for the extraction of vegetable oil from cucumeropsismannii.The functional properties analysed for the purpose of characterization include: Refractive index , density viscosity, iodine value, peroxide value, saponification value and free fatty acid . The refractive index of the varieties of oil was 1.43776 and 1.45469 for hexane and ethanol respectively, the density values (0.8928g/cm^3) for hexaneand (0.9328g/cm^3) for ethanol, the viscosity of(0.4914cst)for hexane and (0.9895cst) using ethanol. These results showed that using Hexane as solvent produced more oil than using ethanol. Qualitatively ethanol moved better. Both oils produced met the recommended WHO standard.

Keywords: CucumeropsisMannii, Ethanol,Hexane, Production, Solvent

INTRODUCTION

Cucumeropsismannii (melon seed) belongs to the family of Cucurbitaceae which has a tremendous genetic diversity, extending to vegetative and reproductive characteristics. They thrive in tropical, subtropical, arid deserts and temperate locations, (Ng, 1993).Melon is an annual, herbaceous, monoecious plant with a non-climbing creeping habit. After planting, they completely cover the soil surface within 3 weeks and flowering starts. The oil is extracted from the seed, and is popularly called ‘Melon’ a name widely used throughout West Africa. The crop had been in cultivation for at least 4000 years mainly for seed Schippers. The crop does well on a sandy free chaining soil. It can also be planted as an intercrop with crops like maize, okro, cassava, and yam; because they are weed suppressors. When planted, it can be harvested between two and half or three months and with good management there

can be a seed yield of 350 – 400kg per hectare. (Achigan-Dako, *et al*, 2008). Analysis made on melon by (Olaofe,*el al.*, 1994) indicates that melon seed consists about 50% oil by weight 37.4% of protein, 2.6% fibre, 3.6% oil, 6.4% moisture.Vegetable oil are oils extracted from natural seeds such as linseed oil from flax seed, fruits or nuts of plants and cottonseed oil from cottonseed by the use of hydrocarbon solvents.

Basically, among the oil seeds, the commercial interest are linseed, soyabeans and castor beans which have an approximation of 33-43%, 18-22% and 48-53% oil contents, respectively.

Industrial, vegetable oil is used for the production of soap, detergent, skin products, candles, perfumes and cosmetic products which are very important product used for several purposes (Obasi, 2012). Over these years, there is a continuous decline in the availability and affordability of vegetable oil as a result of an increase in the demand which makes the conventional source of vegetable oil insufficient. Hence, this research seeks for an alternative and more productive route for the production of vegetable oil to meet the growing demand for industrial application.

The aim of the study is to produce vegetable oil from melon seed (melonitoo seed) by the process of extraction.

The objectives of the Study include; Characterize the physio-chemical properties in melon seed; Carry out the experiment of the extraction. Analyse batch process in refining of vegetable oil; Determine the percentage solvent recovery and Compare extraction efficiency of Hexane and Ethanol.

Experimental Procedure

The extraction of oil was done in the following steps: Drying, Milling, Density Test of the Solvent, viscosity test, pH test, Reflux extraction and solvent recoveryProcedures. The thermometer was filled with heated or chilled water and a stirrer was used to achieve a

desired temperature.The temperature of the bath was to adjusted to the desired temperature by heating or adding ice water. The mixture was stirrer to maintain uniform temperature. At a constant temperature, all fluid were allowed to flow out by lifting up the closing ball valve until the tip of the level indicators just touch the surface. $50cm^3$ receiver was placed under the opening,by the use of stop watch, the time which it took to flask up $50cm^3$ under the opening, was measured.

Density and Specific Gravity

The densities of solids and the liquids were determined. Every essential oil has a density. This physical property is used to evaluate the quality of substances. The density of oil reveals the yield and quality of the oil. It is temperature dependent; the higher the temperature, the lower the density.Although density and specific gravity are general physical characteristic used in the classification of fats and oils, neither is highly definitive for characterization except for a few high -density oils like castor or hydrogenated castor oils. Equation (2.1) is used to determine the SG of the sample.

$$SG=\frac{\text{Density of substance}}{\text{Standard density of water}} \qquad 2.1$$

Methods of obtaining density and specific gravity

Hydrometer method

Pycnometer method(specific gravity bottle or density bottle

Pycnometer Method

Procedure

Density bottle was weighed and measured using the weighing balance. The bottle with liquid was measured and recorded. The spills were cleaned off to avoid error.The weights were compared and recorded

Refractive Index

Refractive index shows the proportion of the speed of light in vacuum to the speed of light in the oil, it is commonly known as the proportion between the sine of the point of refraction when beam of light of a known wave-length (typically 589.3 mu, the mean of the d-lines of sodium) goes from air into the oil, it is useful for identification purposes and purity, and also for observing the progress of reaction, such as catalytic hydrogenation and isomerisation .It is been excessively used in analysing binary esters.

Procedure for Refractive Index

The lamp standing behind the refractometer was switched on and the prison box was opened by releasing toggle on the right-hand side and swung to the left. The two prisons were cleaned with acetone and cotton wool and 3-5 drops of the oil to be examine were placed onto the fixed prison and the apparatus was closed. With the eye on the upper telescope the control knob was turned in front until the field was divided into two(light and dark field). The boarder line between the fields may appearcolored due to dispersion of the lamp light. The color was eliminated by rotation of the dispersion drum (second knob) until the field appeared of good contras and free from color neither red nor blue. The boarder line was set exactly on the intersection of the eye piece. The scale reading was observed in the lower telescope and the fourth number behind the point was interpolated.

Chemical Property Analysis

Determination of Free Fatty Acid(FFA)

their Free Fatty Acid is a significant subjective parameter as far as oil quality is concerned. Since fats and oils contain some degree of free unsaturated fat (FFA), there will consistently be an expansion in sharpness with time during vehicle and capacity. The benefit of fat and oil depends, in some regard, on the measure of thefree unsaturated fat which creates. The physical and substance properties of fat and oils are basically controlled by the unsaturated fat synthesis of triglycerides.

Procedure:

2.5g of oil sample was weighed into a conical flask and 100ml of neutralized alcohol was added to it. Two drops of phenolphthalein were added, and titration was carried out with 0.1N of NaOH until a pink color change was observed. The titrate value was recorded for calculations.

Determination of Hydrogen Ion Concentraction (pH)

The pH of oil determines its relative Acidity or Alkalinity.There are two (2) methods that could be used to determine the pH of solution. Electrometric method (i.e. using pH meter) andcolorimetric method (i.e using litmus paper).

Procedure

The apparatus was sampled and assembled. The pH meter was turned on and allowed to warm for15minutes. The electrode was standardized using standard buffer pH 4or 7

100ml of the oil measured in a measuring cylinder into a 250ml beaker. The electrode was immersed into the oil and allowed the lower part of electrode to reach the bottom of the beaker. The reading was taken and recorded.The electrode was removed and cleaned with distilled water.

Determination of Saponification value of oil

Saponification is the quantity of milligrams of potassium dioxide required to saponify 1g of fat under the condition indicated. It is a proportion of the normal sub-atomic weight (or chain length) of all the unsaturated fats present in the oil.

Procedure

4g of oil was measured into a conical flask containing 50ml of alcohol potash.

The flask was connected to a heater and heated for 1hour while shaking.After 1 hour it was removed and 2 drops of phenolphthalein was added and pink colour emerged. This was titrated using 0.5N hydrochloric acid till the pink color disappeared.The blank (without oil) was done in the same manner with distilled water. The following equation is used to determine the Saponification of oil

$$Sy = \frac{(\text{titration of black} - \text{titration of sample}) * N * 56.1}{\text{weight of sample}}$$

Where,

N=normality of HCl

Determination of Peroxide Value of The Oil

Peroxide value decides all substances regarding miliequivalent of peroxide per 100 grams of test, which potassium iodide under the state of the test(AO.C.S.1986). Its significance is that it demonstrates the extent of deterioration in palm oil (oil and fats). A higher value indicates poor oil quality and favors high free fatty acid (FFA). The peroxide values elucidate the extent of deterioration of oil and likewise give a clue on FFA.

Procedure

About 20ml of oil was measured into 250ml conical flask. About 50ml of acetic and chloroform solution and swirl was added. Then about 0.5ml of potassium iodide was added and shaken vigorously for 1minute. About 30ml of distilled water was added and titrated with sodium thiosulphate until yellow color disappeared. Thesame process was repeated for blank.

Determination of Iodine Value of the Oil

Iodine value is a proportion of the all numberof the two-fold bonds present in fats and oils.It is communicated as the quantity of grams of iodine that will respond with the twofold securities in 100grams of fats or oil. It very well may be resolved in fats and oils with thermometric titration, by dissolving a gauged test in a non-polar dissolvable and after that including frigid acidic corrosive.

Procedure

5g of oil was measured into a 500ml flask. About 20ml of carbon tetrachloride(CCl_4) was added into the flask containing the oil; then 25ml of wiji"s reagent solution was also added

9g of iodine in 1litre of glacial acetic acid was prepared, and 10ml of iodine acetic acid solution was addedintothe 500ml flask. This content was shaken vigorously for about 1 minutes.The flask was then allowed to rest for 30minutes in a dark locker.After which 20ml of potassium iodide (KI) solution followed by 100ml of distilled water were added into the flask.The solution was titrated against 0.1N sodium thiosulphate (Na_2SO_3) and 0.5ml of starch solution was added after first titration. A second titration was done with 0.1N (Na_2SO_3).

RESULTS AND DISCUSSION

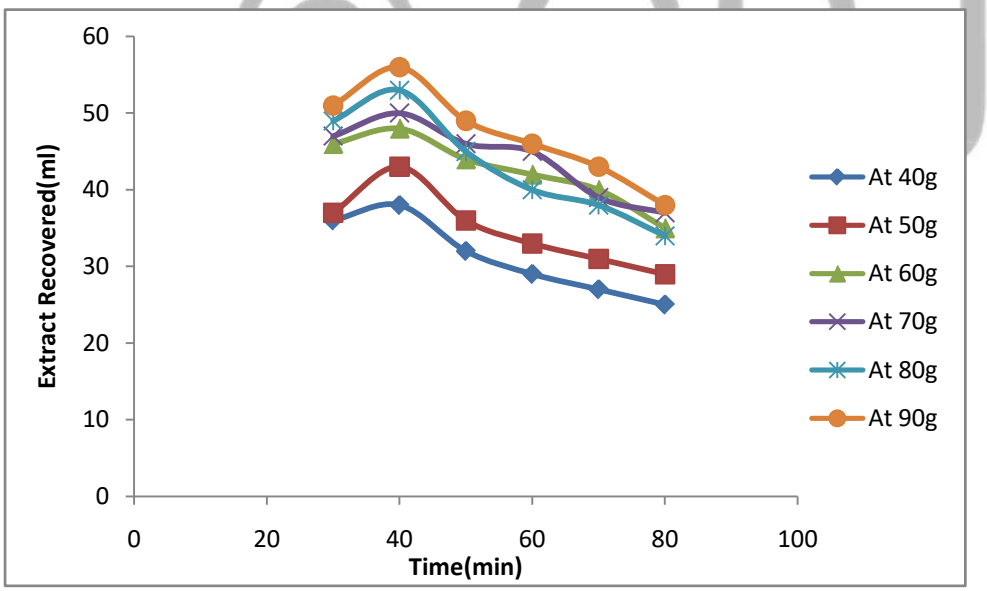

Figure 1: Graph of extract recovered using Hexane at 65^{o}C Versus Time for Concentration of Nutrients

Figure 1, shows the extracts recovered with time for different nutrient weight. From this plot it is seen that there is a decrease in the volume extracted with time for the different weight of sample. This decrease can be attributed to evaporative loss of oil due to increased residence time within the extractor.From the plot, it is also shown that the higher the weight of sample, the greater the extract recovered. The maximum extract recovered was 56ml from 90g of the

sample, while the least 38ml was for the 40g of the sample. Hence the volume of extract recovered is directly proportional to the weight of sample used.

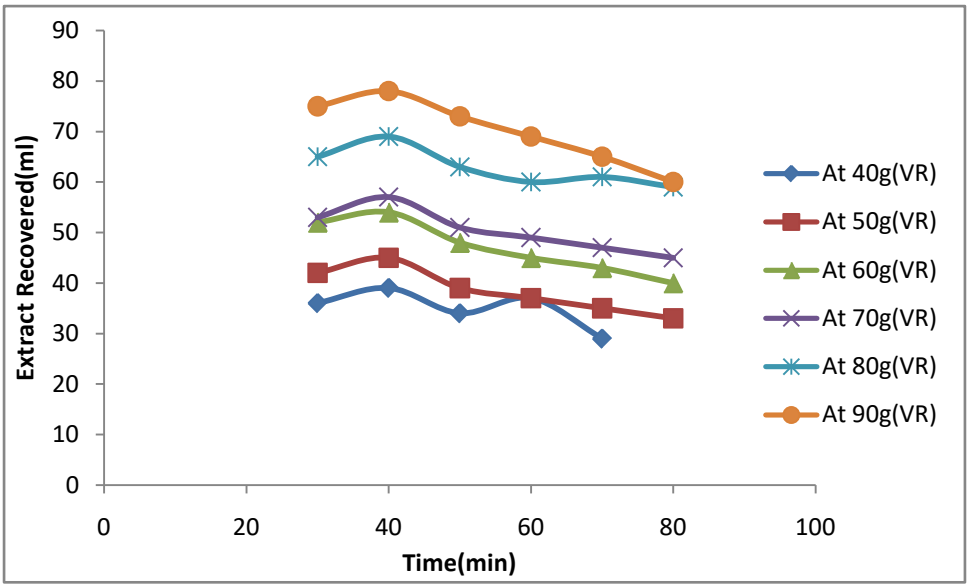

Figure 2: Graph of extract recovered using Hexane at 70^{o}C Versus Time for Concentration of Nutrients

Figure 2, shows the extracts recovered with time for different nutrient weight. From this plot it is seen that there is a decrease in the volume extracted with time for the different weight of sample. This decrease can be attributed to evaporative loss of oil due to increased residence time within the extractor.

From the plot, it is also shown that the higher the weight of sample, the greater the extract recovered. The maximum extract recovered was 78ml from 90g of the sample, while the least 39ml was for the 40g of the sample.

Hence the volume of extract recovered is directly proportional to the weight of sample used.

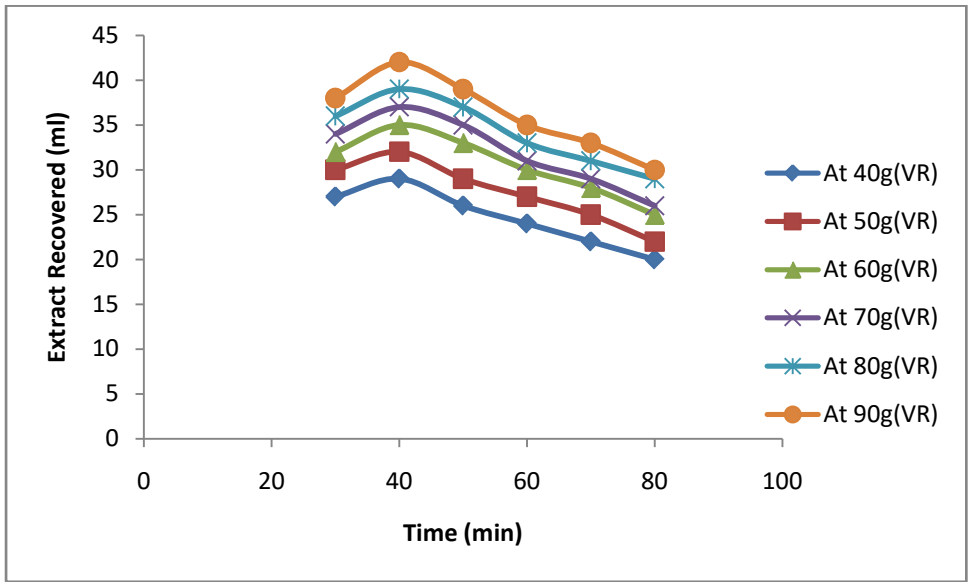

Figure 3: Graph of Extract Recovered using Hexane at 85^{o}C versus Time for Concentration of Nutrients .

Figure 3, shows the extracts recovered with time for different nutrient weight. From this plot it is seen that there is a decrease in the volume extracted with time for the different weight of sample. This decrease can be attributed to evaporative loss of oil due to increased residence time within the extractor.From the plot, it is also shown that the higher the weight of sample, the greater the extract recovered. The maximum extract recovered was 42ml from 90g of the sample, while the least 39ml was for the 40g of the sample.Hence the volume of extract recovered is directly proportional to the weight of sample used.

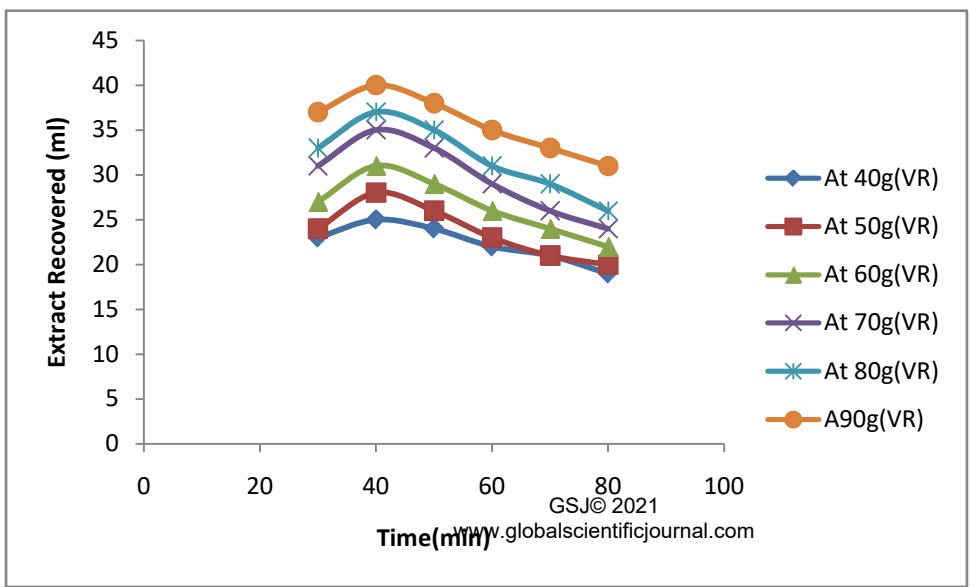

Figure 4: Graph of Extract Recovered using Hexane at 90^{o}C versus Time for Concentration of Nutrients .

Figure 4, shows the extracts recovered with time for different nutrient weight. From this plot it is seen that there is a decrease in the volume extracted with time for the different weight of sample. This decrease can be attributed to evaporative loss of oil due to increased residence time within the extractor.From the plot, it is also shown that the higher the weight of sample, the greater the extract recovered. The maximum extract recovered was 40ml from 90g of the sample, while the least 25ml was for the 40g of the sample.Hence the volume of extract recovered is directly proportional to the weight of sample used.

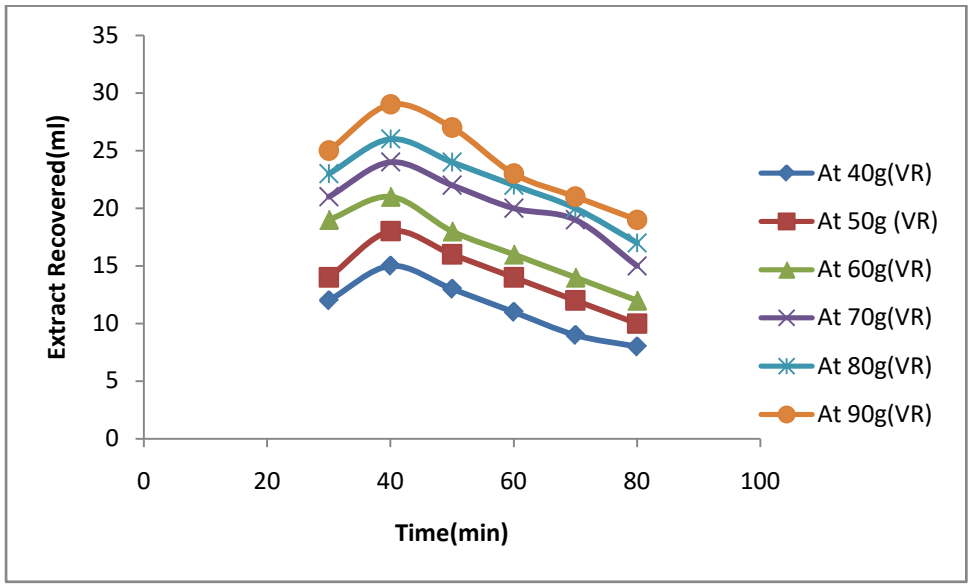

Figure 5: Graph of Extract Recovered using Ethanol at 65^{o}C versus Timefor Concentration of Nutrients .

Figure 5, demonstrates the relation of the functional parameter of the extract recovered.This figure shows that extract recovered varies with time at constant weight of the sample. Increase in extract recovered was observerd within 65^{o}C with increase in time within the range of >0 to< 40minutes. And a decrease in volume of extract was also observed at thesame 65^{o}C. The variation in the volume of extract recovered can be ascribed to variation in time of extraction.

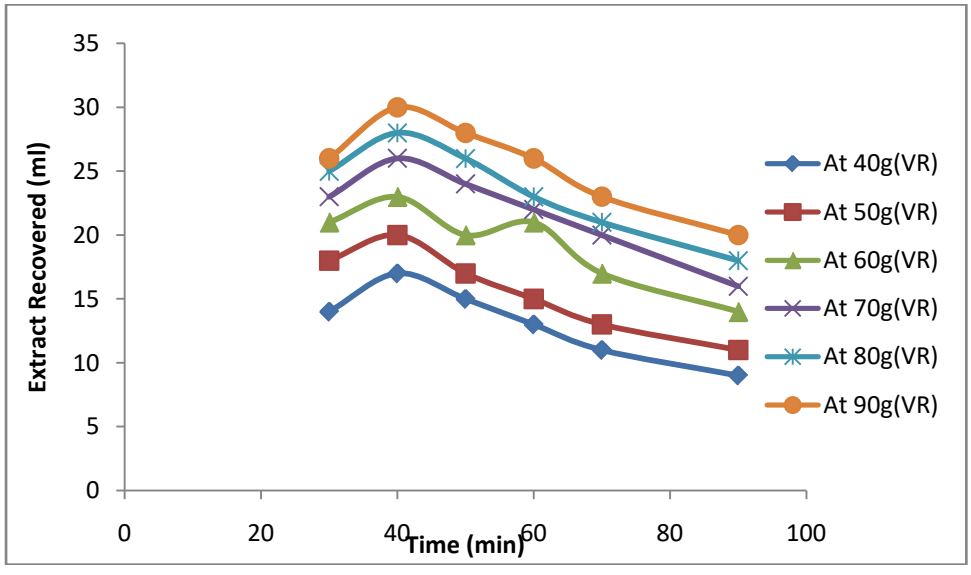

Figure 6: Graph of Extract Recovered using Ethanol at 70^{o}C versus Time for Concentration of Nutrients .

Figure 6, demonstrates the relation of the functional parameter of the extract recovered. This figure shows that extract recovered varies with time at constant weight of the sample. Increase in extract recovered was observed within 70^{o}C with increase in time within the range of >0 to< 40minutes. And a decrease in volume of extract was also observed at the same 70^{o}C. The variation in the volume of extract recovered can be ascribed to variation in time of extraction.

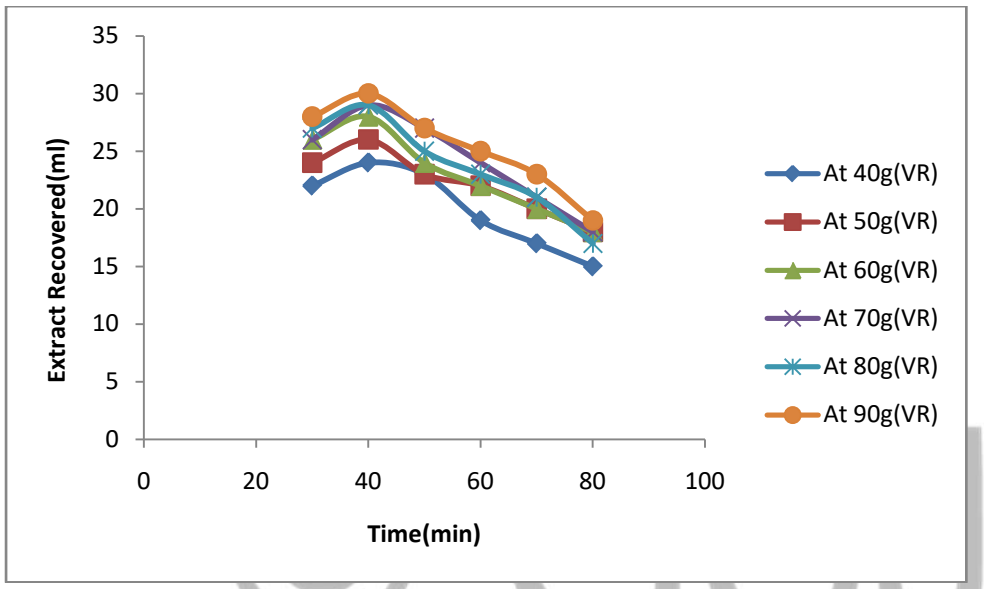

Figure 7: Graph of Extract Recovered using Ethanol at 85^{o}C versus Time for Concentration of Nutrients .

Figure 7, demonstrates the relation of the functional parameter of the extract recovered. This figure shows that extract recovered varies with time at constant weight of the sample. Increase in extract recovered was observed within 85^{o}C with increase in time within the range of >0 to< 40minutes. And a decrease in volume of extract was also observed at the same 85^{o}C. The variation in the volume of extract recovered can be ascribed to variation in time of extraction.

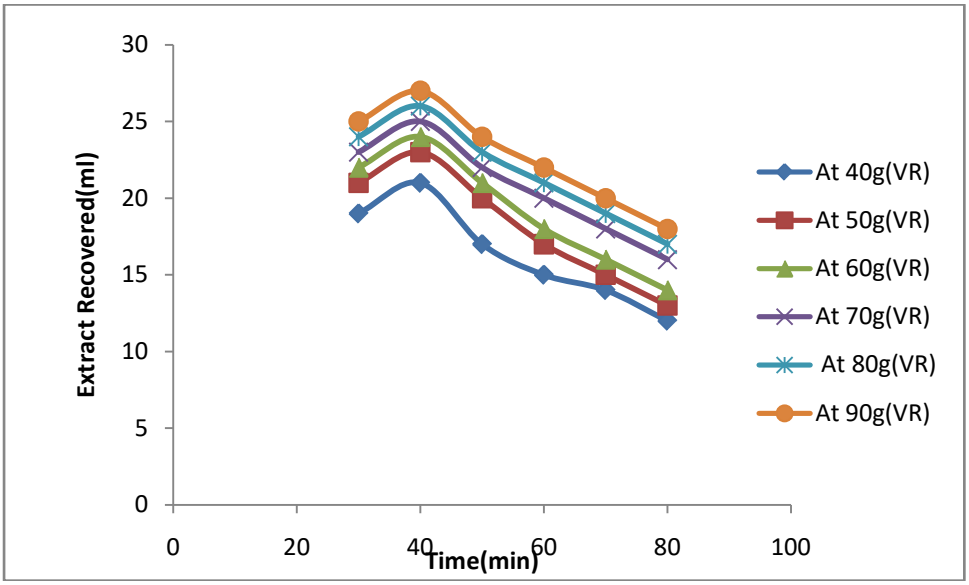

Figure 8: Graph of Extract Recovered using Ethanol at 90^{o}C versus Timefor Concentration of Nutrients .

Figure 8, demonstrates the relation of the functional parameter of the extract recovered.This figure shows that extract recovered varies with time at constant weight of the sample. Increase in extract recovered was observed within 90^{o}C with increase in time within the range of >0 to< 40minutes. And a decrease in volume of extract was also observed at the same 90^{o}C. The variation in the volume of extract recovered can be ascribed to variation in time of extraction.

CONCLUSION

Physical and Chemical analysis of the vegetable oil from cucumeropsismannii (melon-seed) show that the vegetable oil has valuable components that are very important and useful in several purposes including human being, livestock(animals) and also industrially.This work also involved the determination of physical properties of the vegetable oil of plant such as refractive index, density and viscosity, also the chemical properties such as iodine value, acid value, saponification value and free fatty acid of the oil. The physicochemical characteristics

of the oil constitute useful referencepoints of comparison being used as quality standards for commercial transactions.

From GC and MS Analysis result it was also observed from fatty element dictated that the oil contain Undecylenic acid 19.72% for oil recovery from Hexane and 13.44% for oil recovery from Ethanol. These powerful undecylenic acid is used in production of Nylon and active ingredient in medication for skin infection and relieve itching and burning. It is also a precursor in the precursor in the manufacture many pharmaceutical, personal hygiene products, cosmetics and perfumes. Other noticeable substances which seems very vital in human body are Myristic acid 11.38 % Plamiticoleic acid 18.43% for oil recovery from Hexane and Myristic acid 9.55 % Plamiticoleic acid 10.77% for oil recovery from Ethanol were observed to be present essential oil. These substances are very useful in the production of drugs to that cures stroke and fungal infections.

The results above indicate the value of refractive index of these 1.43776 for Hexane and 1.45469 for Ethanol. Density (g/cc) 0.8928 for Hexane and 0.9328 for Ethanol. The Viscosity (cst) was 0.4914 for Hexane and 0.9895 for Ethanol.These results showed that using Hexane as solvent produced more oil than using ethanol. Qualitatively ethanol move better.

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