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UTILIZING ORANGE PEELS FOR ESSENTIAL OIL PRODUCTION

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ABSTRACT

Presently, in Nigeria orange peels are discarded as wastes after consumption of edible parts of orange fruits. However, the country depends on imported essential oil for use in industries for manufacture of products ranging from food, to beverage, cosmetics and pharmaceuticals. This research work was carried out to demonstrate the utilization of orange peel in the production of essential oils by employing different extraction methods. The methods considered in this work were steam distillation, water distillation and solvent extraction. For solvent extraction, Design Expert 7.0 was used to design experimental runs because of its relative popularity among the extraction methods. After the extraction, the oil was analysed to determine its physical and chemical properties. From the results obtained, it was discovered that the orange peels could give the maximum yields of essential oil to be 4.40%, 3.47% and 2.536% when steam distillation, water distillation and solvent extraction were employed, respectively, and that was an indication that the highest yield of essential oil was given by steam distillation among the methods considered. It was also discovered from the analysis of variance carried out on the results of experimental design done for solvent extraction method that a modified cubic model was able to represent the extraction process well because the model was obtained to be significant, and its square of correlation coefficient was reasonably high. Furthermore, the analysis of variance of the developed model revealed that the significant factors of the process were extraction time and extraction temperature. The characterization of the extracted oil gave its physical and chemical properties values that indicated that it could be used for production of other valuable products in different process industries.

Keywords: orange peels, essential oil, extraction, design expert, experimental design.

1. INTRODUCTION

Essential oil is obtained from plant material which is held within certain part of the plant or specific part of the plant cells; it may be from leaves, seeds, peels or stalks, depending on the species. The methods used for obtaining essential oil include hydro-distillation, or solvent extraction, supercritical fluid extraction, cold pressing, microwave extraction [1].

Citrus fruits belong to six genera (Fortunella, Eremocitrus, Clymendia, Poncirus, Microcitrus, and Citrus), which are native to the tropical and sub-tropical regions of Asia, but the major commercial fruits belong to genus *Citrus*. The genus citrus includes several important fruits such as oranges, mandarins, lime, lemons and grape fruits [2, 3]. The essential oil is present in the fruit's peel in great quantities. The citrus essential oil is a mixture of volatile compounds and mainly consists of monoterpene hydrocarbon [4, 5].

Essential oils are mixtures of over a hundred compounds that can be approximated into three fractions: terpene hydrocarbons, oxygenated compounds and non-volatile compounds. The terpene fraction can constitute from 50 to more than 95% of the oil [2]. Essential oil from citrus is a large type of natural flavours and fragrances which is popularly used in food industries, daily chemical products and health care field. The citrus species are potential sources of variable oil which might be utilized for edible and other industrial purposes. Essential oils are broadly used as pharmaceutical components, in nutrition supplements and for cosmetic industry and aromatherapy [6]. The exact function of essential oils in a plant is unknown, it may be to attract insects for pollination, or to repel harmful insects or it may be simply a metabolic intermediate [3].

In Nigeria and other parts of the world, citrus is cheaply available, and it, thus, serves as a major source of vitamins in diets. Citrus fruit and its juice have several beneficial, nutritive and health properties. They are rich in vitamins especially ascorbic and folic acids. Over the last decade, many other virtues and medicinal benefits of citrus fruits have been discovered beside their anti-scurvy properties [7].

The current annual world production of citrus fruit is approximately 110 million tons. In Nigeria, about 930,000 tons of citrus fruits are produced annually from an estimate of 3 million hectares [3, 8]. In 1993, the world sales value of fragrance and flavours was 19Billion USD [9]. Out of this amount, Nigeria didn't earn anything, but rather spent about $14million on importation of flavours, fragrances and essential oils between June and December 1994 [10].

Currently, Nigeria's local production of essential oil is insignificant, so nearly 100% of the essential oils used by our local industries are imported. Research statistics from the Raw Materials Research and Development Council (RMRDC) indicates a local demand of over 100,000kg, annually; a figure that could be met through local production efforts [3].

Generally, the waste disposal problem from juice producing industries and fruits such as orange peels causes environmental pollution. In order to reduce this problem, the waste (peels of citrus fruits) can serve as raw material

for the extraction of essential oils needed for various domestic and industrial uses.

According to the information gathered from the literature, Kabuba [11] investigated the parameters required for steam extraction of obtaining essential oil and found out that increase in temperature and pressure, which were observed to be significant in essential oil production, were making the yield of the essential oil to increase. Ramgopal *et al*. [12] used orange peel to obtain essential oil D-Limonene using simple distillation and discovered from the work that the volume of oil extracted was increasing with time of heating in all cases. Also, they found out that the maximum yield of oil was obtained when distilled water was used as the solvent. Nautiyal and Tiwari [13] carried out their work on extracting oil from orange peel using SC-CO_2 at pressures ranging from 8-15 MPa and temperatures ranging from 28-60 ℃ to study the quality, quantity and compositions of the oil. The quality of the oil extracted in the work was analysed with the aid of Gas Chromatograph and Capillary Gas Chromatograph. It was discovered from the work that supercritical carbon dioxide was able to extract the oil, which was light pale yellow in colour, containing all the low volatile fractions and oil obtained. Ahmad *et al.* [14] applied a novel closed system extraction method for obtaining essential oil by investigating its impact on the yield and the physical characterization of the oil and was able to conclude that the technique gave a better oil with less impurity. Mercy *et al*. [15] employed improved steam distillation, where the orange peels were preheated before subjecting to distillation to oil extraction from orange. It was discovered from the work that the preheating enhanced the yield of the oil obtained. Shakir and Salih [16]carried out the extraction of essential oil from orange (*Citrus sinensis*), lemon (*Citrus limon*) and mandarin (*Citrus reticulata*) peels using steam distillation and microwave assisted steam distillation to study the effect of extraction conditions (weight of the sample, extraction time, and microwave power, citrus peel type) on oil yield. The extracted essential oil was analysed by Gas Chromatograph. It was found out that microwave-assisted extraction was better than ordinary steam distillation in terms of rapidity, energy saving and yield. Sikdar *et al*. [17]investigated the use of orange (*Citrus sinensis*) peels for the extraction of citrus oil employing improved steam distillation, where the orange peels were preheated before subjecting to distillation. The extracted citrus oil was found to be composed of about 95% d-limonene, which has many applications ranging from food flavouring agents to cosmetics.

The literature review carried out has revealed that the research into essential oil extraction is becoming serious owing to the usefulness of the oil. In order to contribute our quota to this area of research, this work has been carried out to investigate the yield of essential oil from citrus peel, which is presently discarded in Nigeria as waste, using different methods of extraction. Conventional experimental design was used to obtain steam and water distillation experimental runs. While central composite method of RSM was employed to design the n-hexane extraction of the orange oil. In all the methods the effects of extraction time and temperature variations on the yield of orange oil processed orange peels were investigated.

2. METHODOLOGY

The extraction of essential oil was accomplished in one of the laboratories of Abubakar Tafawa Balewa University, Bauchi, Nigeria using water distillation, steam distillation and solvent extraction methods on a laboratory scale. The orange peels used were acquired from Wunti Market, Bauchi, Bauchi State, Nigeria. After the orange peels were obtained, they were washed, cleaned with water and sundried for five days. The dried peels were then pulverized before being used in each of the extraction methods investigated in this work.

Outlined below are the procedures used for the different oil extraction methods employed in this research.

2.1 Steam distillation procedure

150 g of the prepared orange peels were introduced into the distillation flask, which was connected to a round bottom flask containing water. The flask was connected to a condensing unit with its tubing. The set-up of the distillation unit, which had heating mantle, is shown in Figure-1.

The essential oil was extracted with the distillation set-up using steam as it was percolating through the peels. The recovered mixture of oil and water were allowed to settle and the oil was withdrawn. After the steam distillation process, the product, which was a mixture of water and oil, was collected and separated using separating funnel. The essential oil settled on the top layer and the water was in the bottom layer of the funnel. The mixture was separated until negligible amount of water was left with the oil.

Figure-1. Steam distillation set-up.

Figure-2. Water distillation set-up.

2.2 Water distillation procedure

150 g of the orange peel powder was weighed using digital weighing balance and then transferred into a round bottom flask with large amount of water added to cover the peels. The flask was connected to the still column which was connected to the condenser, as shown in Figure-2. The steam generated from the water being heated extracted the essential oil which was subsequently condensed as part of steam as it was passing through the condenser. The distillate, a mixture of water and oil, was then collected and poured into the separating funnel, where the mixture was separated into two layers (oil at the upper layer and water at the lower layer). Water was later separated and the oil was collected in a bottle. The experimental run was carried out at different temperature intervals, and the volume of oil was taken over a constant interval of time.

2.3 Solvent extraction procedure

To carry out this procedure, the ground peels were sieved using a standard 0.6 mm particle size sieve. Thirteen (13) experimental runs were generated using central composite design with two factors, having five centre points, which was designed with the aid of Design Expert 7.0[18]using the design parameters given in Table-1. The two factors considered in the experimental design were time and temperature. After the experimental design, the extraction was carried out using normal hexane as the extraction solvent. 10 g of the sample was used for each run, and it was executed using Soxhlet apparatus that is set up as shown in Figure-3. In the Soxhlet apparatus, the solvent in the round bottom flask was heated from the heating mantle to become evaporated and got condensed down through the sample where it was able to extract the oil along, thereby, giving a mixture of oil and solvent, which was later separated.

Table-1. Experimental design parameters for the solvent extraction system.

Factor	A	B
Name	Time	Temperature
Units	Min	^{o}C
Type	Numeric	Numeric
Low Actual	63.43	40
High Actual	176.57	70
Low Coded	-1	-1
High Coded	1	1
Mean	120	55
Std. Dev.	44.38	11.77

Figure-3. Solvent extraction setup.

2.4 Calculation of oil yield

The yield of the oil extracted using each of the three methods of extraction was calculated using Equation (1).

$$\%Yield = \frac{Weight\,of\,oil\,extracted}{Weight\,of\,sample\,used}100\% \quad (1)$$

2.5 Characterization of the essential oil

After the oil was extracted, it was characterized in order to be sure that the extracted liquid was actually the oil. Outlined below are the tests carried out on the oil.

2.5.1 Sensory analysis of the essential oil

Sensory analysis was carried out on the oil to determine its physical properties. This involved sense of sight, smell and touch.

2.5.2 Determination of solubility of the essential oil in water

A few drops of the oil was added to a test tube containing little amount of water. The test tube was stirred thoroughly with a stirring rod. Two separate phases were observed.The insolubility of the oil in water was inferred from that operation.

2.5.3 Determination of specific gravity of the oil

A clean and dry bottle was weighed using a weighing balance. Distilled water was poured into the bottle and weighed. In the same manner, the same volume of oil was poured into the same bottle and weighed. The specific gravity was calculated as the ratio of weight of oil to that of water [19] as given in Equation (2).

$$Oil\ specific\ gravity = \frac{Weight\,of\ particular\ volume\,of\,oil\ extracted}{Weight\,of\,equal\,volume\,of\,water} \quad (2)$$

2.5.4 Determination of saponification value of the essential oil

Saponification value, being the weight of potassium hydroxide expressed in milligrams that is required to saponify 1 g of oil was also determined in this work. To carry out this, 2 g of the oil was weighed into a 200-ml conical flask unto to which 50ml of 0.5 M of KOH was added. The resulting mixture was refluxed for 30 minutes, followed by addition of 3 drops of phenolphthalein indicator, and it was titrated against 0.5 M HCl until coloration disappeared. This procedure was repeated without the oil and the titre value was determined from the blank value [19]. The saponification value was calculated using Equation (3).

$$Saponification\ value = \frac{(t_2 - t_1) \times 28.1}{W} \quad (3)$$

where, $t_1$is the blank titre value, $t_2$is the sample titre value and W is the weight of the sample.

2.5.5 Determination of acid value of the oil

To determine the acid value of the extracted oil, 2 g of the oil sample was weighed into a conical flask containing 50 ml of isopropyl alcohol. 3 drops of phenolphthalein indicator were added to the mixture. The resulting mixture was titrated against 0.1 M NaOH [19], and Equation (4) was applied to calculate the acid value of the oil.

$$Acid\ value = \frac{(5.61 \times titre\ value)}{Weight\ of\ sample} \quad (4)$$

2.5.6 Determination of ester value of the oil

Ester value, which is defined as the number of milligrams of potassium hydroxide required to saponify the fatty acid esters in one gram of the oil, was also determined for the oil extracted in this work. It was obtained as the difference between the saponification value and the acid value of the oil [19] as given in Equation (5).

$$Ester\ value = Saponification\ value - Acid\ value \quad (5)$$

2.5.7 Determination of density of the oil

The density of the oil extracted was determined by weighing an empty beaker and recording its value. Thereafter, essential oil was poured into the beaker and the weight was taken. The density of the oil was thus calculated using Equation (6).

$$Density = \frac{Weight\ of\ oil\ sample}{Volume\ of\ oil\ in\ the\ beaker} \quad (6)$$

2.5.8 Determination of free fatty acid of the oil

1 g of the essential oil was poured in a beaker and warmed; 25 ml of methanol was added to the sample and stirred thoroughly followed by 2 drops of phenolphthalein indicator and a drop of 0.14 N NaOH solution. The mixture was titrated against NaOH solution until a light pink colour which persisted for about 1 minute was observed. The end-point was recorded and used to calculate the free fatty acid from Equation (7) [19].

$$FFA = \frac{(titre\ value \times N \times 28.2)}{Weight\ of\ sample} \quad (7)$$

Where FFA denotes the free fatty acid and N is the normality of the base.

2.5.9 Determination of iodine value of the oil

1 g of the essential oil was weighed and added to 10 ml of CCl_4. The entire content was dissolved in 10 ml of wij's solution by swirling. It was kept in a dark place for 30 minutes. The solution was titrated against sodium thiosulphate with starch as an indicator. The same procedure was repeated for blank titration [19]. The iodine value was calculated using Equation (8).

$$Iodine\ value = \frac{(B - S) \times N \times 12.69}{W} \quad (8)$$

where B is the blank titre value, S is the sample titre value and the weight of the sample is denoted as W.

2.5.10 Determination of peroxide value of the oil

30 ml of acetic acid/ chloroform was measured into a flask containing 2 g of the oil sample. A 0.5ml saturated solution of potassium iodide was then added, followed closely by the addition of 30 ml of distilled water. The flask content was titrated against 0.1 M sodium thiosulphate until the resulting colour disappeared. Thereafter, 0.5 ml starch indicator was added, and the titration was continued till an end-point was observed. A blank titration experiment was also performed [19], and the peroxide value was calculated using Equation (9).

$$Peroxide\ value = \frac{(S - B) \times 0.1 \times 1000}{W} \quad (9)$$

where B is the blank titre value, S is the sample titre value and the weight of the sample is denoted as W

3. RESULTS AND DISCUSSIONS

The results obtained from the work carried out on the extraction of essential oil from orange peels, using the three methods considered (water distillation, steam distillation and solvent extraction processes), are outlined thus.

3.1 Results for water distillation

Water distillation extraction method of obtaining essential from orange peels was carried out at a temperature of 95^{o}C in this work, and the results obtained from this were as shown in Figure-4. It can be seen from the figure that at no oil was obtained from the extraction from the initial time up till about 80 min. Thereafter, the amount of oil given with time was found to increases significantly. It was also observed from the figure that the yield of oil obtained when the extraction time was 200 min was 2 ml/150 g peel.

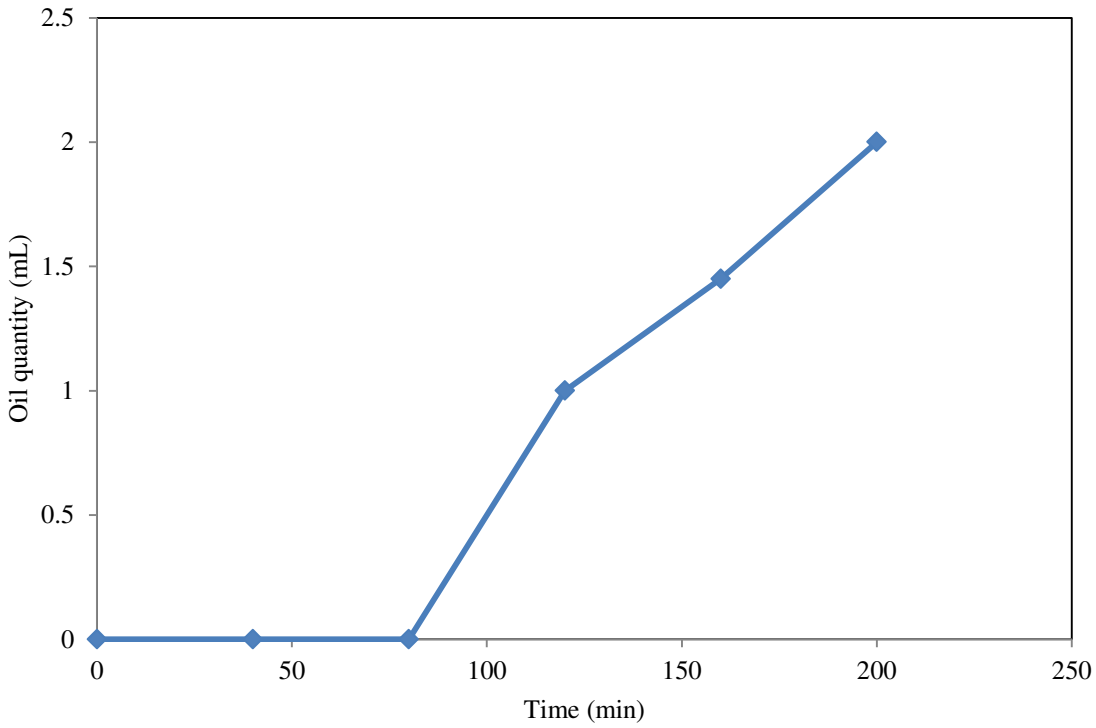

Figure-4. Response of water distillation extraction process at 95°C.

Carrying out the same water distillation procedure of the essential oil extraction at a temperature of 100°C gave the results that are shown in Figure-5. From the results given in Figure-5, it was noticed that oil was produced by the extraction at the temperature of 100°C from the initial time of the process, and, even, the amount of oil given was observed to keep on increasing with increase in extraction time. For this process, which was carried out at 100°C, the yield of oil obtained when the extraction time was 200 min was obtained to be 5.2 ml / 150 g peel amounting to 3.47% oil yield.

Analysing the results given in Figures 4 and 5, it can be seen that temperature is one of the factors affecting extraction process because the amount of oil obtained at the temperature of 95°C was found to be different from the amount given when the extraction temperature was 100°C. It has also been observed that the high temperature could favour extraction process because the oil given by the extraction process carried out at 100°C was found to be higher than that given when the temperature was set to 95°C.

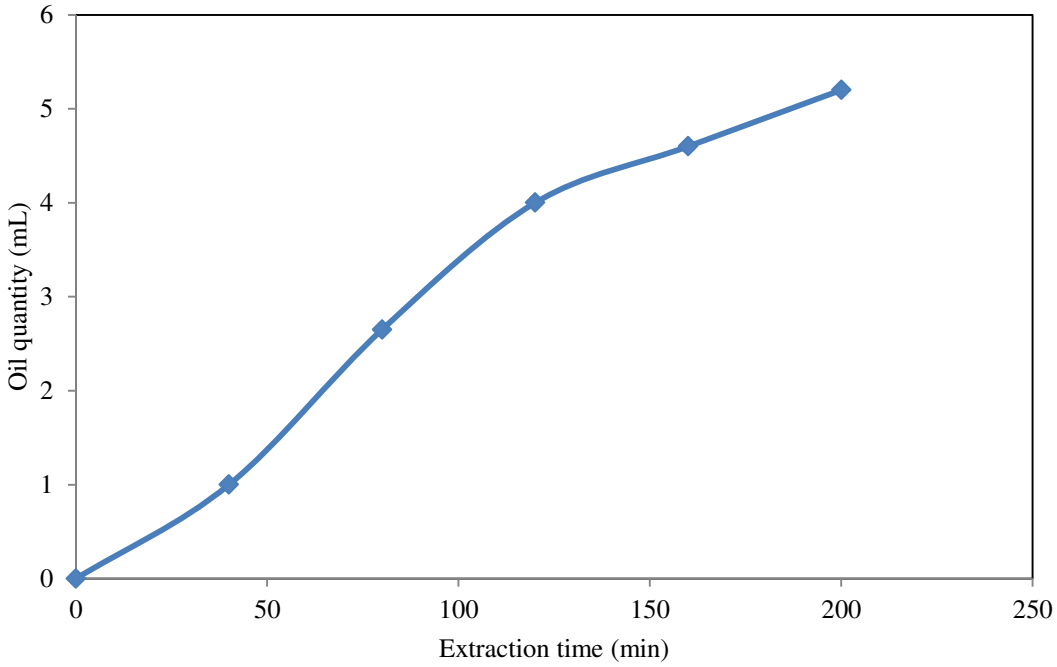

Figure-5. Response of water distillation extraction process at 100°C.

3.2 Results for steam distillation

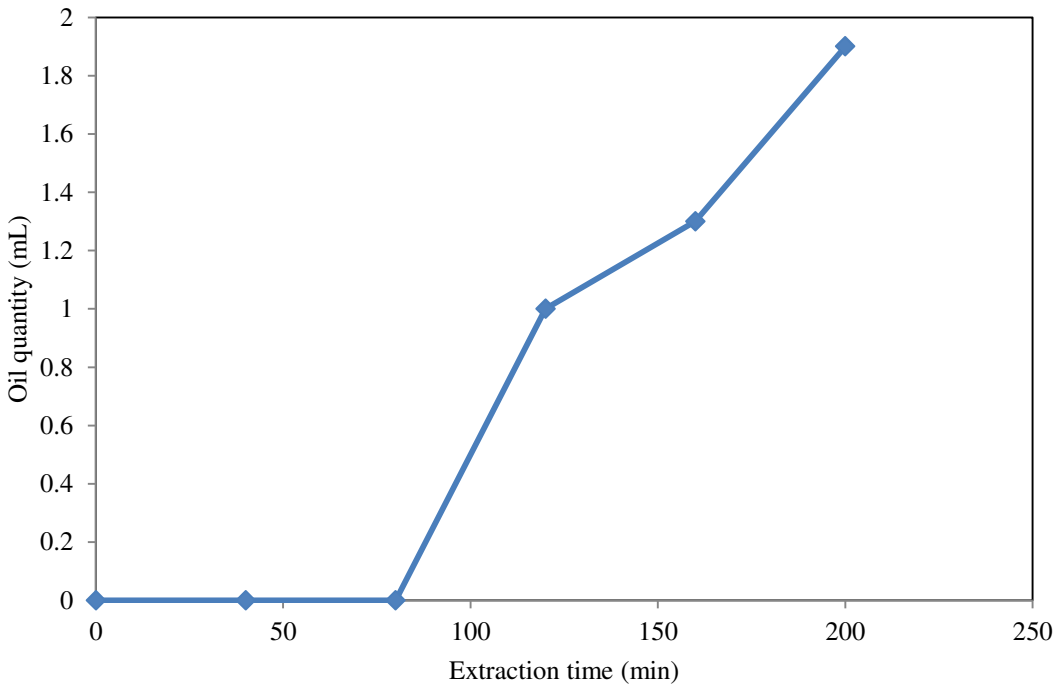

Figure-6. Response of steam distillation extraction process at 95°C.

Shown in Figure-6 is the variation of the amount of oil obtained from the process when the extraction was carried out using steam distillation at 95°C. From the figure, it can be seen that no oil was extracted from the system within the first 80 min, but after that, extraction of essential oil started, and it kept increasing with as the time of extraction was also increasing. According to the figure, the yield of the oil obtained when the extraction time was 200 min was about 1.9 ml / 150 g of the peels.

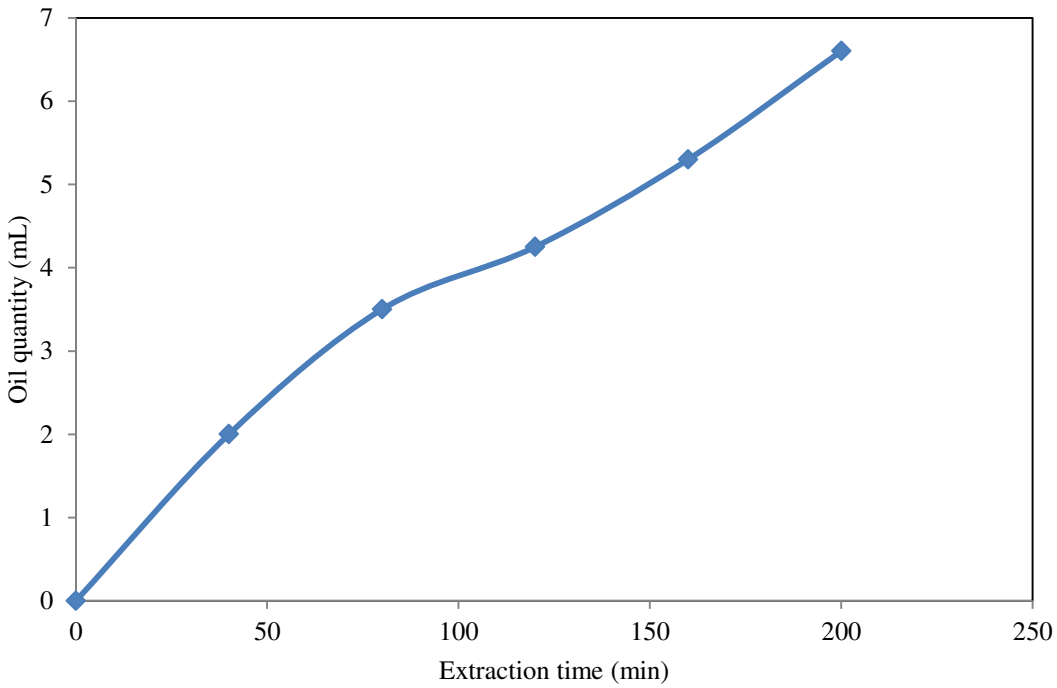

Figure-7. Response of steam distillation extraction process at 100°C.

Also, when the extraction was carried out at a temperature of 100°C, steam distillation extraction was observed to response by giving essential oil from the initial period when the extraction was commenced. Just as it was discovered in the case of the water distillation, the yield of oil obtained in this case of 100°C was more than that of 95°C because the yield of the oil given by this case was at the extraction time of 200 min was about 6.6 ml / 150 g of the peels (4.4% oil yield). This case has also indicated the dependency of extraction process on temperature as the amount of oil was found to vary as the temperature of the extraction process was varied.

Actually, based on the information obtained from the literature, hydro-distillation extraction is, generally, dependent on the temperature and pressure of the process [11]. This can be explained by considering the fact that at low temperatures, steam goes up into the plant material slowly, and its pressure build up is not sufficient enough to extract the oil out of the peels matrix. However, if left for a longer period of time, the oil will eventually break out of the peels matrix and, thus, be extracted. That is why it was noticed from the extraction process carried out that at a temperature of 95°C, there was no yield initially, but after some time, the steam being formed rather more rapidly, and pressure being obviously higher, the oil extraction began, and it was continuous thereafter.

3.3 Results for solvent extraction

Solvent extraction, being one of the most popular methods in use, was taken with more seriousness in this work by designing the experiments to be carried out with it with the aid of Design Expert, the results of which are shown in Table 2. The results given in Table 2 shows that the variations in the factors chosen as the independent variables of the process were having effects on the yield of oil obtained from the extraction of orange peels. The change in either time or temperature was found to affect the yield of the oil. The recurring time and temperature, that is, the centre points, gave almost the same percentage yield of oil.

Furthermore, the results obtained when analysis of variance (ANOVA) was carried out on the data generated after the experiment for solvent extraction using Design Expert 7.0 were as given in Table 3. The ANOVA was carried after modifying the cubic model equation (Equation 10) of the process, which had R-squared value of 0.8136. From the ANOVA, considering 95% confidence level, it was found that the entire model was significant with p-value of less than 0.05. The results also showed that both temperature and time were affecting the yield of the essential oil yield because they were found to be significant with the p-value of each of them also being less than 0.05 based on 95% confidence level.

$$Oil\ \ yield\ \ = 11.9593 - 0.1566A - 0.2672B + 0.003021AB + \Lambda$$
$$\Lambda + 0.0006512A^2 + 0.001032B^2 - 0.00001199A^2B \quad (8)$$

Table-2. Experimental data and response obtained from the solvent extraction process.

Run	A: Time (min)	B: Temperature (^oC)	Oil yield (%)
1	176.57	70	2.54
2	120	55	1.35
3	63.43	40	1.49
4	120	55	1.52
5	120	55	1.31
6	176.57	40	2.36
7	120	55	1.49
8	200	55	1.54
9	120	55	1.43
10	40	55	0.66
11	120	76.21	2.38
12	120	33.79	0.85
13	63.43	70	1.19

Table-3. Outputs of analysis of variance.

Source	p-value
Model	0.0480
A- Time (min)	0.0123
B- Temperature (^oC)	0.0201
AB	0.5058
A^2	0.8491
B^2	0.1275
A^2B	0.0571

3.4 Characterization of the essential oil

Moreover, the extracted essential oil was analysed to determine its physical and chemical properties, and given in Tables 4 and 5 are the results obtained from the analyses for the physical and the chemical properties respectively. As can be seen from the Table-4, the oil was obtained to have virtually yellow colour with tangy smell, and it was observed to be insoluble in water. The density of the oil was estimated to be 0.86 g/cm^3 (see Table 4 for details).

Table-4. Physical properties of essential oil from orange peel waste.

Parameter	Value
Colour	Yellow to orange
Odour	Fresh to tangy smell
Solubility	Insoluble in water
Density (g/cm^3)	0.86
Specific Gravity	0.843

Table-5. Chemical properties of essential oil from orange peel waste.

Parameter	Value
Saponification value	43.71
Acid value	3.88
Ester value	39.83
Free fatty acid value	1.94
Iodine value	98.16
Peroxide value	12.06

The values obtained for the chemical properties of extracted essential oil are given in Table-5. Those chemical properties determined were the saponification value, the acid value, the ester value, the free fatty acid value, the iodine value and the peroxide value of the oil, as shown in Table-5.

Comparing the specific gravity of the extracted oil with the literature, it was discovered that the value of the specific gravity obtained for the extraction essential oil of this work compared well with the value obtained in the work of Barkatullah *et al*. [20]. At least, this was found to be an indication that the extracted material from the orange peels used was an essential oil type.

4. CONCLUSIONS

The results obtained from the extraction and the characterization of the essential oil from orange peels have shown that the maximum yield of essential oil obtained from the orange peels used in this work were 4.4%, 3.47% and 2.536% when the methods employed were steam distillation, water distillation and solvent extraction respectively, indicating that steam distillation was able to give the highest yield of essential oil among the methods considered. Though, steam distillation gave the maximum,

solvent extraction is still more promising as the yield was achieved at lesser temperature (70^{o}C) which capable of giving oil of better quality. The experimental design and data analysis carried out on solvent extraction method made it known that a modified cubic equation could be used as a model to represent the extraction process with significance and a reasonable value of square of correlation coefficient (R-squared value).It was shown by the analysis of variance of the developed model that time and temperature are very significant factors on the yield of the oil extracted in the process. The physical and chemical properties values obtained from the characterization of the oil revealed that it could be used in different process industries for production of other valuable products. It was palpable from the values of the oil yields obtained using the given quantity of orange peels that large amount of the raw material, which was a waste, would be required for large scale extraction of the oil, thus, making this process an advantage for the environmental and waste management sectors of the community.

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