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Extraction of oil from selected plants using Response Surface Methodology [RSM]

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Abstract
This study involves the extraction of oil from three sources: orange peel, guava leaves, and cassia fistula leaves using Soxhlet apparatus. The variables considered in this study were time of extraction and sample weight. Minitab statistical software was used to randomize the runs. The combination of operating parameters that gave the optimum yield for the three sources were identified. The regression equation for each source was reported. The coefficient of determination (R^2) value for orange, guava leaves and cassia fistula extract were 99.51%, 99.90%, and 99.77% respectively. This shows that the model is a good prediction tool for extraction of oil from these sources. Based on the R^2 values guava leaves (99.90%) gave the highest prediction accuracy followed by cassia fistula (99.90%), with orange leaves having the lowest R^2 value (99.77%) among the three sources considered.

Key words: RSM, Extraction, Soxhlet, Yield, Cassia fistula.

1. Introduction
Plant essential oils have been widely used for many years due to their antimicrobial properties in foods and pharmaceutical products, some have been used as aromatic plant since ancient times [1-3], while high repellence against arthropod species and various types of insects was discovered in others [4-6]. It has long been observed that plants’ flowers, roots, leaves and seeds contain some active ingredients which are known as Essential oil. Some are odoriferous, some are volatile and Ethereal (being extractable with ether an organic solvent). Parts of the naturally occurring organic substance present in the plant are the essential oils which are the secondary substances in plants and have been found to be of great importance to the human life. Odour of the active ingredients in plants may serve as attractant of insects in some species, thereby promoting pollination. While in others, they may serve as repellent to insects thereby reducing the possibility of being attacked by the pest [2, 7].

Cassia fistula has numerous medicinal properties like astringent, cooling, laxative, febrifuge, tonic, purgative, anthelmintic, emetic, antiperiodic, febrifuge, diuretic, depurative, carminative, calming, diuretic and ophthalmic. Utilized in numerous therapeutic treatments: skin ailments, consuming sensations, syphilis, bubbles, sickness, ringworm affection, colic, dyspepsia, clogging, diabetes, strangury, cardiovascular issues, dry hack, bronchitis, jungle fever, stiffness, fever, uncleanness, Stomach issue, aggravations and discontinuous fever. Guava or Psidium guajava is a low evergreen tree with spreading branches and square, downy twigs, is a native of tropic America. Guava is a tropical and semitropical plant. It is known for
its edible fruit. It is known usually to be in the backyards, its branches are crooked bringing opposite leaves. *Psidium guajava* is a tree which is 20 feet high having also spreading branches. It is not hard to know when you see it because of its smooth, thin, copper-colored bark that flakes off, reviewing the greenish layer under and also because of the attractive bony part of its trunk which may after some time reach a diameter of 10 inch [8].

In the fruit processing plant, the management system is challenged by the waste they generate such as peels and skins. Orange peel is a major waste that has a substantial burden on the environment; hence it is necessary to find a feasible way to dispose of the peels to have a positive environmental impact or turn them into useful products [9]. Recent discovery by researchers revealed how to curb environmental pollution caused by various materials especially papers, nylon and bottle. These papers can be recycled so that they can be re-used instead of being wasted [10, 11]. Orange peel is a waste which can serve as an alternative source of energy when pectin is extracted from it [12].

Response surface methodology (RSM) has been employed to model and optimize the extraction of oil from orange peel, guava and Cassia fistula leaves which can be used for several microbial and insecticidal activities [13]. Mathematical model is regarded as a decision tool that assists decision makers in effectively dealing with complex issues such as oil spillage on soil surfaces [14-17].

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**Figure 1:** Cassia fistula tree

**Figure 2:** Guava leaves

**Figure 3:** Orange peel
2. Methodology

Materials and Method

Sample collection: *Cassis fistula* and Guava leaves were collected from the Covenant University Farm. Oranges were purchased from an open market.

Raw materials preparation

Guava and *Cassia* leaves: Both leaves were plucked, washed thoroughly with distilled water, air dried under shade for 14 days and then powdered with the help of Binatone electric blender.

Orange peel: Orange peel sample was prepared according to [12].

Extraction: Extraction of crude oil from the three materials was done using [18-21] method.

3. Results and Discussion

Table 1 presents the results obtained from the study carried out on the extraction of oil from orange peel, guava leaves and *Cassia fistula*, using Soxhlet apparatus and n-hexane as the solvent for extraction. Two variables considered are extraction time and weight of seeds. The effect of these two variables on the yield was also discussed.

| Runs | Time (hr) | Weight (g) | Experimental yield (%) | Predicted Yield (%) |
|------|-----------|------------|-------------------------|---------------------|
|      |           | Orange peel | Guava leave | Cassia fistula | Orange peel | Guava leave | Cassia fistula |
| 1    | 4         | 10         | 23.00       | 29.50          | 24.00       | 22.75       | 30.01       | 23.69       |
| 2    | 6         | 20         | 16.50       | 22.00          | 13.50       | 16.02       | 22.22       | 13.40       |
| 3    | 6         | 10         | 26.00       | 30.50          | 23.00       | 26.42       | 30.17       | 22.69       |
| 4    | 2         | 20         | 15.50       | 22.20          | 15.00       | 15.19       | 22.19       | 15.15       |
| 5    | 4         | 10         | 23.00       | 30.00          | 23.00       | 22.75       | 30.01       | 23.69       |
| 6    | 4         | 30         | 9.33        | 14.50          | 8.66        | 8.87        | 14.41       | 8.52        |
| 7    | 4         | 30         | 9.00        | 14.40          | 8.66        | 8.87        | 14.41       | 8.52        |
| 8    | 6         | 30         | 12.00       | 14.30          | 12.00       | 12.54       | 14.27       | 12.02       |
| 9    | 6         | 20         | 16.50       | 22.30          | 13.00       | 16.02       | 22.22       | 13.40       |
| 10   | 2         | 10         | 25.50       | 30.10          | 29.00       | 25.58       | 29.84       | 28.94       |
| 11   | 2         | 20         | 15.00       | 22.20          | 15.50       | 15.19       | 22.19       | 15.15       |
| 12   | 4         | 20         | 11.50       | 22.20          | 12.00       | 12.36       | 22.21       | 12.14       |
| 13   | 4         | 20         | 12.50       | 22.20          | 12.50       | 12.36       | 22.21       | 12.14       |
| 14   | 4         | 20         | 12.00       | 22.20          | 12.00       | 12.36       | 22.21       | 12.14       |
| 15   | 2         | 30         | 11.66       | 14.50          | 9.00        | 11.70       | 14.54       | 9.27        |

Effect of weight and time on the yield of orange extract

From Table 1, it can be seen that the optimum yield of orange peel extract was 26% at extraction time of 6 hours and sample weight of 10 g. Surface and contour plots in Figure 4 shows that as the sample weight increases, the yield also increases. It was observed that at low extraction...
time, there was lower yield of oil extracted as compared with higher extraction times. The regression equation which shows the effect of extraction time and sample weight on the orange extract is displayed in Eqn. 1.

\[
\text{Orange peel extract (\%)} = 52.20 - 6.284 \times \text{Time (hr)} - 2.075 \times \text{Weight (g)} + 0.8116 \times \text{Time (hr)} \times \text{Time (hr)} + 0.03454 \times \text{Weight (g)} \times \text{Weight (g)}
\]

(1)

The fitness of the model was determined by the $R^2$ value. It was reported that for a good fit of the model the $R^2$ should be greater than 0.8 [22, 23]. $R^2$ value was 99.51 % which indicated that the sample variation of 99.51 % of the extraction process was analyzed by the model. $R^2$ adjusted was also high at 99.31 %, which supports a high significance of the model [22]. The predicted $R^2$ value was 98.93 % which is in a reasonable agreement with the adjusted $R^2$ and thus indicates the efficacy of the model as a good representation of the interaction between the variables considered. Figure 5 shows a plot of experimental against predicted values which validates the efficiency of the model in predicting the yield of the orange extract.

**Figure 4:** The surface and contour plot showing interactive effect of the variables on orange yield
From Table 1, it can be seen that the optimum yield of guava extract was 30.5% at extraction time of 6 hours and sample weight of 10 g. Surface and contour plots in Figure 6 shows that as the sample weight increases, the yield also increases. This was similar to what was observed for orange peel extract. It was observed that at low extraction time, there was a low yield of oil extracted as compared with higher reaction time. The regression equation which shows the effect of extraction time and sample weight on the guava extract is displayed in Eqn. 2.

\[
Orange\ peel\ extract\ (\%) = 37.182 + 0.156 \times Time\ (hr) - 0.7500 \times Weight\ (g) \\
- 0.00750 \times Time\ (hr) \times Weight\ (g)
\]

The fitness of the model was determined by the $R^2$ value. It was reported that for a good fit of the model the $R^2$ should be greater than 0.8 [22, 23]. $R^2$ value was 99.90% which indicated that the sample variation of 99.90% of the extraction process was discussed by the model. $R^2$ adjusted was also high at 99.87%, this support a high significance of the model [22]. The predicted $R^2$ value was 99.71% which is in a reasonable agreement with the adjusted $R^2$ and thus indicate the efficacy of the model as a good representation of the interaction between the variables considered. Figure 7 shows a plot of experimental against predicted values which validates the efficiency of the model in predicting the yield of the guava extract.

Figure 5: Plot of Experimental yield against predicted yield of orange extract.

Effect of weight and time on the yield of guava extract

From Table 1, it can be seen that the optimum yield of guava extract was 30.5% at extraction time of 6 hours and sample weight of 10 g. Surface and contour plots in Figure 6 shows that as the sample weight increases, the yield also increases. This was similar to what was observed for orange peel extract. It was observed that at low extraction time, there was a low yield of oil extracted as compared with higher reaction time. The regression equation which shows the effect of extraction time and sample weight on the guava extract is displayed in Eqn. 2.
Figure 6: The surface and contour plot showing interactive effect of the variables on guava yield.

Figure 7: Plot of experimental yield against predicted yield of guava extract

Effect of weight and time on the yield of *Cassia fistula* extract

From Table 1, it can be seen that the optimum yield of *Cassia fistula* extract was 29.00 % at extraction time of 2 hours and sample weight of 10 g. Surface and contour plots in Figure 8 shows that as the sample weight increases, the oil yield decreases. This can be due to the fact that a higher solvent volume and temperature was required to get a higher yield. A greater amount of solvent may be required as the sample weight was increased. The regression equation that shows the effect of extraction time and sample weight on the guava extract is displayed in Eqn. 3.
The fitness of the model was determined by the $R^2$ value. It was reported that for a good fit of the model the $R^2$ should be greater than 0.8 [22, 23]. $R^2$ value was 99.77% which indicated that the sample variation of 99.77% of the extraction process was represented by the model. $R^2$ adjusted was also high at 99.65%, this support a high significance of the model [22, 24]. The predicted $R^2$ value was 99.27% which was in a reasonable agreement with the adjusted $R^2$ and thus indicate the efficacy of the model as a good representation of the interaction between the variables considered. Figure 9 shows a plot of experimental against predicted values which validates the efficiency of the model in predicting the yield of the *Cassia fistula* extract.

\[
\text{Cassia fistula extract (\%)} \\
= 62.42 - 6.948 \times \text{Time (hr)} - 2.7925 \times \text{Weight (g)} \\
+ 0.5325 \times \text{Time (hr)} \times \text{Time (hr)} + 0.03960 \times \text{Weight (g)} \times \text{Weight (g)} \\
+ 0.11250 \times \text{Time (hr)} \\
\times \text{Weight (g)} \quad (3)
\]

Figure 8. The surface and contour plot showing interactive effect of the variables on *cassia fistula* yield.
4. Conclusion

The use of Soxhlet apparatus was applied in the extraction of volatile components from three sources which are orange, guava leaves and Cassia fistula. A maximum orange peel extract (26.00%) was attained at extraction time of 6 hours and sample weight of 10 g. Increase in the sample weight and maintaining the extraction time shows that there was a decrease in the amount of orange peel extract. This can be due to the amount of solvent volume used in this extraction process. The maximum guava leaf extract was also obtained at the same condition of extraction time (6 hours) and sample weight (10 g). Maximum guava leaf extract was 30.50%, this also shows that the highest guava leaf extract was obtained at high extraction time and low sample weight. While the maximum Cassia fistula extract was obtained at a extraction time of 2 hours and a low sample weight of 10 g. It can be inferred for the three sources that as the sample weight increases with low extraction time the amount of extract reduces, this can be due to low contact time of the seeds with the solvent volume used. It can also be attributed to the fact that higher solvent volume should be considered while using a higher weight of seeds. The quadratic model generated for the three sources shows that Minitab 17 is a good optimization tool with $R^2$ values of 99.51%, 99.90% and 99.77% for orange, guava leaves and Cassia fistula extract. Based on the adjusted $R^2$ values of 99.31%, 99.87%, and 99.65% for orange, guava leaves and Cassia fistula respectively it supports a high significance of the model. The predicted $R^2$ values of 98.93% (orange), 99.71% (guava leaves) and 99.27% (Cassia fistula) indicated the model efficiency in the adequate representation of variables among the variable considered.

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