International Journal of Chemical and Biomedical Science
2018; 4(1): 11-15
http://www.aascit.org/journal/ijcbs
ISSN: 2472-9574 (Print); ISSN: 2472-9590 (Online)

American Association for Science and Technology

Extraction, Optimization and Characterization of the Ethiopian *Ruta Chalepensis* Crude Oil

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Keywords
Ruta Chalepensis,
Response Surface Methodology,
Physico-chemical Properties,
Optimization,
Crude Oil

Received: January 3, 2018
Accepted: January 16, 2018
Published: January 25, 2018

Citation
Keresa Defa, Anuradha Jabasingh Sekarathil, Gizachew Shiferaw, Sisay Feleke, Etefa Gurmesa. Extraction, Optimization and Characterization of the Ethiopian *Ruta Chalepensis* Crude Oil. *International Journal of Chemical and Biomedical Science.* Vol. 4, No. 1, 2018, pp. 11-15.

Abstract
In this study extraction, optimization and characterization of *Ruta chalepensis* crude oil was investigated. The oil was extracted in a soxhlet apparatus using hexane as a solvent. The process parameters such as extraction temperature, extraction time, and solid to solvent ratio were optimized using the Response Surface Methodology (RSM). Face Centered Cube (FFC) of 3-level-3-factorial experimental design was used to investigate and validate the extraction parameters affecting the yield with a total of 20 experiments of two replicates and six center points. The maximum crude oil yield of 16.68% was obtained at an extraction temperature of 85°C, extraction time of 6 h and the solid to solvent ratio of 13.

1. Introduction

Ruta chalepensis is originally indigenous to the Mediterranean region, Canary Islands, Cape Verde Islands, Sudan, Ethiopia, and Somalia for cooking and medicinal purposes [1]. *Ruta chalepensis* is commonly known as rue and used in the traditional medicine of many countries for the treatment of a variety of diseases [2]. It is used for the treatment of rheumatism, mental disorders, dropsy, neuralgia, antivenom, nervous disorders, convulsions, rheumatism and bleeding disorders [3] [4]. They are characterized by the presence of alkaloids (acridone, quinolone, furoquinolone type alkaloids, and quaternary furoquinolines, furano coumarins) and essential oils [5] [6]. The essential oils from aerial parts of *Ruta chalepensis* plants contain major components including 2-undecanone, 2-nonanone, 2-nonyl-acetate, and 2-dodecanone [5]. However, this plant is not utilized for its full potential for other applications. This partly may be due to lack of adequate research and knowledge on the properties and characteristics of this plant. As a result, this work address the extraction, characterization, and optimization of processing parameters that affects the yield of the crude oil from this plant, which could pave the way for its utilization and wider application at a larger scale.

2. Materials and Methods

2.1. Raw Materials

Ruta chalepensis was obtained from Wando Genet Agricultural Research center (WGARC), Ethiopia. The dirty substances harvested along with all the plant part is removed and washed by the tap water. All the aerial parts of the plant were identified and dried at room temperature for three weeks. The dried aerial parts were reduced in size to the desired particle size of (0.25-0.75mm) by Cross beater Mill.

2.2. Soxhlet Extraction Process

Ruta chalepensis oil was then extracted by Soxhlet apparatus. The work focused on the effect of these parameters, temperature, extraction time, and solid to solvent ratio (SSR) on the yield of the *Ruta chalepensis* crude oil. Extraction was carried out by Soxhlet apparatus [7]. Different SSR of hexane was poured into the round bottom flask. 30g of the sample was placed in the thimble and inserted in soxhlet extractor. The extraction was continued based on the level of the input variables or process parameters selected [8]. At the end of the extraction, the resulting mixture (miscella) containing the oil was heated to recover solvent from the oil using a rotary evaporator under reduced pressure by vacuum pump, and the yield was calculated.

2.3. Soxhlet Extraction Process and Oil Characterization

The optimal extraction conditions for yield of *Ruta chalepensis* crude oil were determined using RSM. The 3-level, 3-factorial Face Centered Cube (FFC) experimental design was used to investigate and validate extraction parameters affecting yield. A summary of the extraction temperature, extraction time, and SSR of 20 experiments with 6 center points were evaluated. Each experiment was carried out in duplicate. The software Design-Expert (Trial Version 8.0.4, Stat-Ease Inc., and Minneapolis, MN, USA) was employed for experimental design, data analysis, and model building. Physical Properties of *Ruta chalepensis* including the moisture content, melting point, pH, specific gravity and the boiling point were determined. Chemical properties of *Ruta chalepensis* crude oil including saponification value, iodine value, peroxide value and acid value were determined [9] [10].

3. Results and Discussion

3.1. Yield of *Ruta Chalepensis* Crude Oil

The yield of crude oil from 20 experimental runs of different combination as per the experimental design were taken as the mean of two replicates plus standard deviations and tabulated in Table 1.

Table 1. *Yield of Ruta chalepensis crude oil extract.*

Run	Temperature (°C) (A)	Time (h) (B)	SSR (C)	First replicate	Second replicate	Yield =μ±sd
1	76.50	4.00	9.00	12.700	10.300	11.500±1.697
2	68.00	2.00	13.00	9.510	11.650	10.580±1.513
3	76.50	2.00	11.00	12.053	12.353	12.203±0.150
4	85.00	4.00	11.00	14.733	11.723	14.493±1.369
5	76.50	4.00	11.00	13.300	12.700	13.000±0.590
6	68.00	4.00	11.00	10.223	12.233	10.973±1.420
7	76.50	4.00	11.00	13.560	11.520	12.540±1.020
8	85.00	6.00	13.00	16.888	16.648	16.768±0.164
9	85.00	6.00	9.00	15.460	14.840	15.150±0.430
10	76.50	6.00	11.00	12.470	14.530	13.500±1.456
11	68.00	6.00	9.00	11.220	8.420	9.820±1.980
12	76.50	4.00	11.00	12.800	13.640	13.220±0.954
13	85.00	2.00	9.00	13.256	12.695	12.976 ±0.397
14	68.00	2.00	9.00	9.520	8.440	8.980±0.764
15	76.50	4.00	11.00	11.561	14.261	12.911±1.090
16	68.00	6.00	13.00	11.786	14.166	12.976±1.190
17	76.50	4.00	13.00	14.902	12.442	13.672±1.379
18	76.50	4.00	11.00	11.156	13.796	12.476±1.320
19	85.00	2.00	13.00	14.500	11.840	13.220±1.881
20	76.50	4.00	11.00	12.020	13.800	12.910±0.890

3.2. Model Equation for *Ruta Chalepensis* Oil Extraction

From the Analysis of Variance (ANOVA) for Response Surface Linear Model, the Values of Prob >F less 0.05 indicate model selection was significant and A, B and C are significant model terms. Values greater than 0.100 indicates that the model terms are not significant. Post ANOVA analysis of the "Pred R-Squared" of 0.9921 is in close agreement with the "Adj R-Squared" of 0.9946. "Adeq precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. In the linear model of this analysis the signal to noise ratio is 105.281 indicates an adequate signal and all the data analysis results show that the data are statistically significant and this model can navigate the design space [11]. Figure 1 shows the effect of processing variables on oil yield. The Linear model equations of factors are can be expressed in two different ways. These are the model equation in terms of the coded variables was

$$\text{Yield} = 12.71+1.84A+1.11B+0.95C$$

Diagnosing of statistical adequacy of the model after post ANOVA analysis is the necessary part before examining the model graphs and optimization.

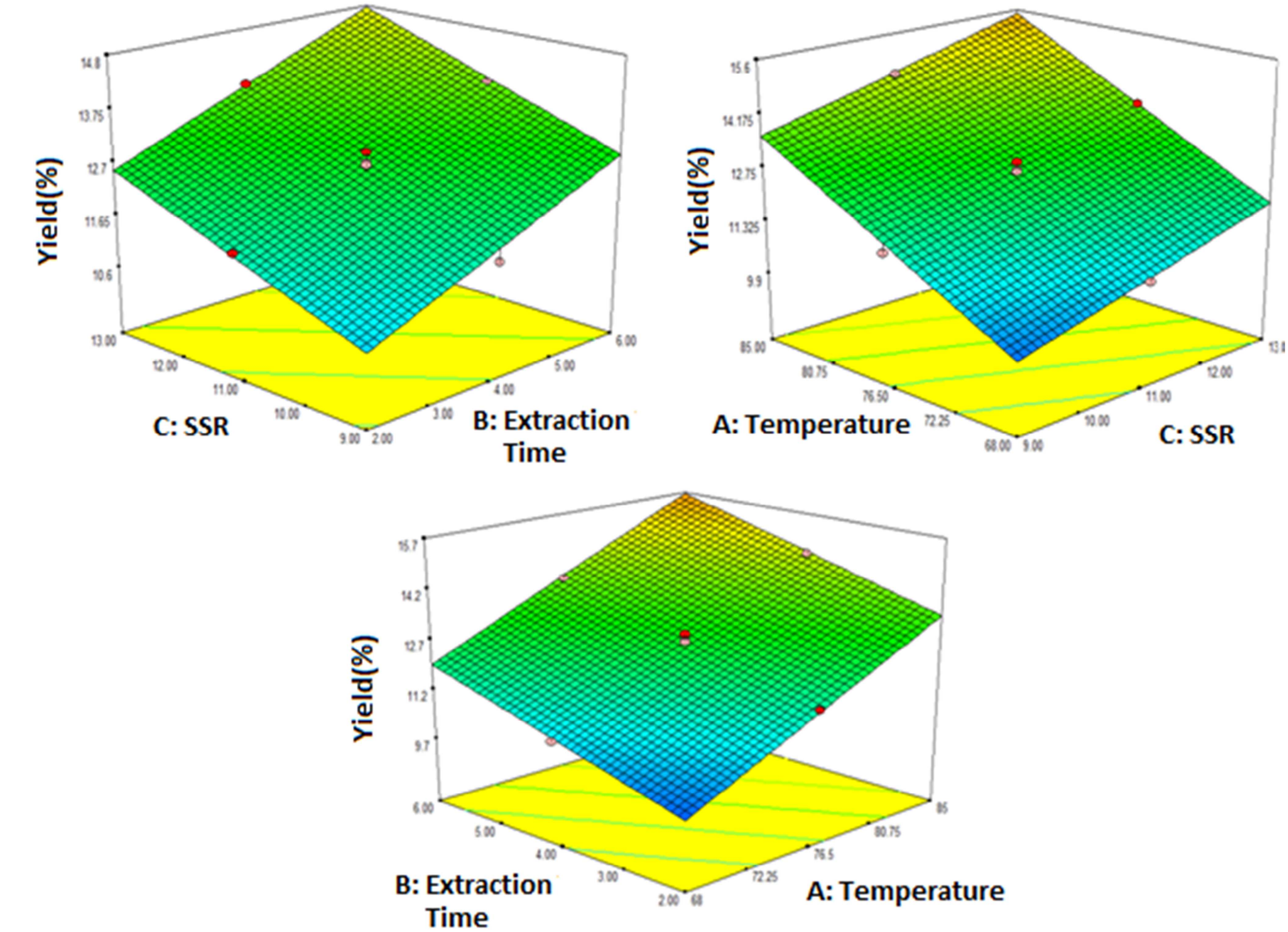

Figure 1. *Effect of processing variables on the yield of crude oil (Response surface plot).*

The yield of *Ruta Chalepensis* crude oil exhibits a linear increasing trend during the temperature and time combination studies, but an increase in the temperature results in a higher increase in the yield rather than the increase in the yield observed during the increase in the extraction time. This could be evidenced from the steepness of the response surface plot of yield with temperature and extraction time, respectively. Similarly, when the effects of experimental parameters of temperature and SSR on the yield were studied, there was a linear increase in the yield of *Ruta Chalepensis* crude oil and the steepness was vivid for the temperature, as a parameter. In all the combinations, an increase in the temperature followed by a clear steepness for the other two variables, shows that temperature highly affects the amount of crude oil produced and it is the most significant factor compared to the other two variables. This fact could be attributed to the oil diffusion coefficient and the oil solubility, which appear to be enhanced during the rise in temperature in the extraction process. In addition, all the parameters were observed to have a positive effect on the response.

3.3. Diagnostic Test for Response

There are different ways of diagnostic tests in Design-Expert. Some of them are Normal probability plot of studentized residuals, Predicted versus actual, studentized residuals versus factor, Box-Cox plot, leverage, etc. The most important diagnostics are the normal probability plot of the residuals and predicted versus actual. In the normal probability plot of studentized residuals (Figure 2a) for the diagnosing of the model adequacy checking, the data points should be approximately linear and a non-linear pattern (look for an S-shaped curve) indicates non-normality in the error term. Likewise, the data points should be linear and all the entire response data should line up on the curve of predicted versus actual plot. Figure 2b showed actual values and predicted values were lined relatively near to the straight line, indicating good fitness of the model. For residuals versus run plot (Figure 2c) all the data points should be connected with one another and should form visible pattern. These plots validate precisely that using linear equation is effective in analysis of response and it is the indication of better fitness of the model to the experimental data. Optimization was carried using the proficiency of 'point optimization' for all variables. The targeted criterion was maximized for the yield of *Ruta Chalepensis* while the factor values were set in their range [12].

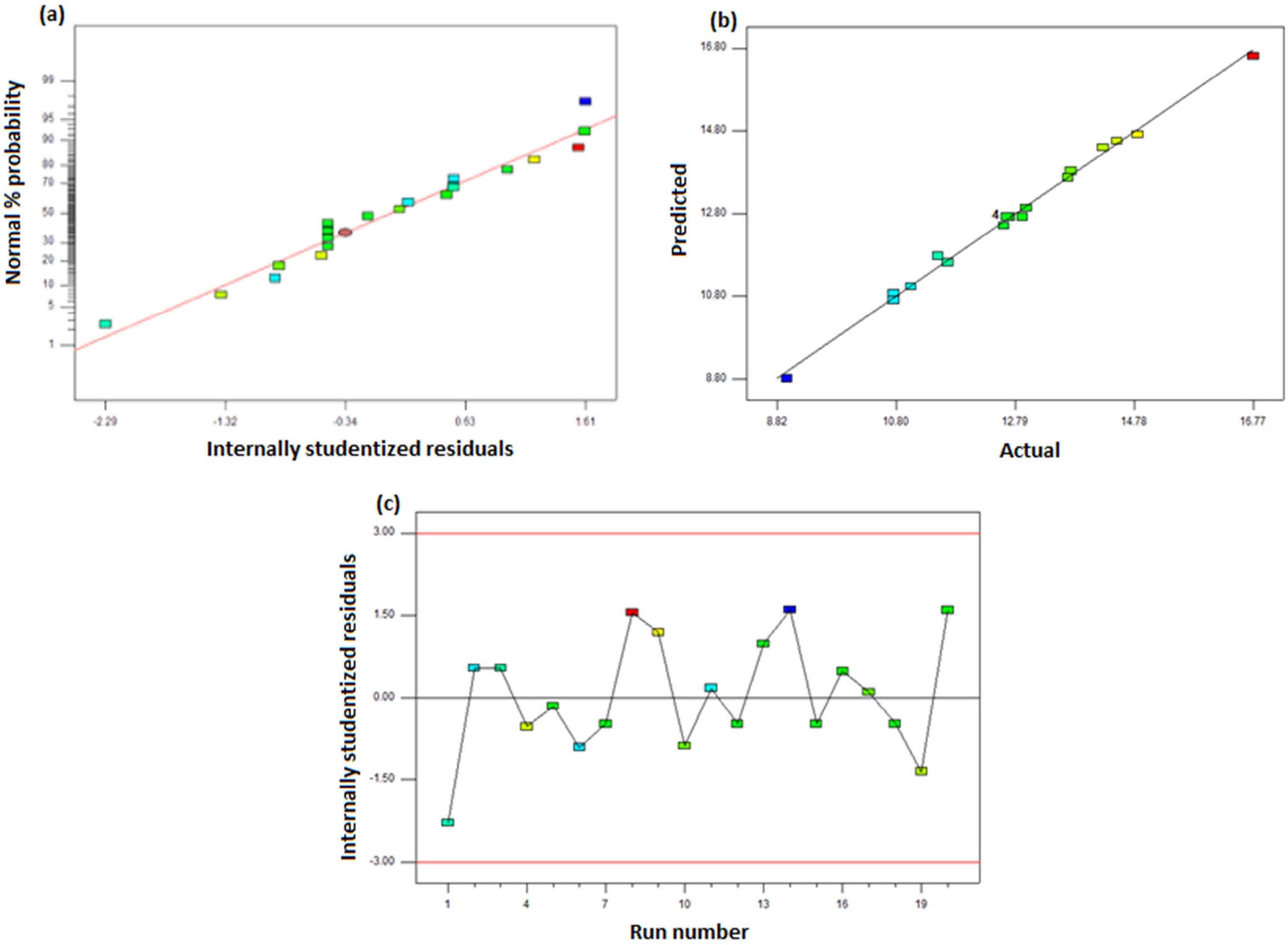

Figure 2. *Model Adequacy checking Diagnostic Test plots.*

3.4. Model Validation

A set of solutions was generated by the software to determine the optimum conditions of the process. These select the values of high composite desirability and maximum response. A combination of factors that simultaneously satisfied the requirements were placed on the ultimate goals of response and all the variables provided the highest composite desirability (0.998). The very small deviation, between the predicted and experimental values of yield in percentage of *Ruta chalepensis* crude oil indicates that the model is suitable and sufficient to predict the crude oil extraction process using the soxhlet extraction in the range of variables studied.

The model predicted 16.605 yield of the oil at an extraction temperature of 85°C, extraction time of 6 h, and solid to solvent ratio of 13. The experimental value obtained in these conditions was 16.768 and is closely in agreement with the result obtained from the model and hence validated the findings of the optimization. The physico-chemical properties of the *Ruta Chalepensis* crude oil during its optimum yield is shown in Table 2. The oil had an acid value of 6.73mg KOH/g of the sample, peroxide value of 5.023 mEq of active O_2/ kg of oil, iodine value of 0.50706 g I_2/100 g of the oil, and Saponification value of 196mg KOH/g of the oil.

Table 2. *The physical properties of Ruta chalepensis crude oil.*

Melting point	50 ±3°C
Boiling point	160±2$^{\circ C}$
PH of the oil	4.496-5.443
Specific Gravity	0.725±0.285
The color of the oil	Dark green
Moisture content	3.28±0.685%
Odor	Pleasant smell

4. Conclusions

This work on the extraction, optimization and characterization of *Ruta chalepensis* oil revealed that the minimum yield of the *Ruta Chalepensis* oil is 8.98 percent by mass obtained at an extraction temperature of 68°C, extraction time of 2 h and solid to solvent ratio of 9. The maximum yield was 16.78 percent by mass found at an extraction temperature of 85°C, extraction time of 6 h and solid to solvent ratio of 13. The optimum value of the process parameters during the soxhlet extraction were temperature 85°C, extraction time 6 h and solid to solvent ratio of 13 for the optimization in the range of the level of the input variables. The physico-chemical properties of the *Ruta Chalepensis* show that the oil is not susceptible to oxidation or oil deterioration and could be further investigated for possible applications.

References

[1] P. C. M Jansen, Spices, condiments and medicinal plants in Ethiopia, their taxonomy and agricultural significance, *Agricultural Research Reports* 906, Centre for Agricultural Publishing and Documentation, Wageningen, Netherlands. 1981, pp 327.

[2] H. D. Neuwinger, *African traditional medicine: a dictionary of plant use and applications*, Medpharm Scientific, Stuttgart, Germany. 2000, pp 589.

[3] J. Mejri, M. Abderrabba, M. Mejri. Chemical composition of the essential oil of Ruta chalepensis L.: Influence of drying, hydrodistillation duration and plant parts. *Ind. Crop. Prod.* 32, 671-673, 2010.

[4] K. R. Kritikar, B. D. Basu, *Indian Medicinal Plants* Vol. 1, 2nd Edn. Allahabad, India, 1984, pp. 452.

[5] G. L. Gamier, Bézanger-Beauquesne, G. Debraux, *Ressources médicinales de la flore française*, 1961.

[6] J. M. Watt, M. G. Breyer-Brandwijk, The *medicinal and poisonous plants of southern and eastern Africa*, 2nd edn, 1962.

[7] F. Chialva, A. Ariozzi, D. Decastri, P. Manitto, S. Clementi, D. Bonelli. Chemometric investigation on Italian peppermint oils. *J. Agri. Food Chem*. 41, 2028-2033, 1993.

[8] M. C. Menkiti, C. M. Agu, T. K. Udeigwe, Kinetic and parametric studies for the extractive synthesis of oil from *Terminalia catappa* L. kernel, *Reac. Kinet. Mech. Cat*. 120, 129-147, 2017.

[9] B. S. Nayak, K. N. Patel. Physicochemical Characterization of Seed and Seed Oil of Jatropha curcas L. Collected from Bardoli (South Gujarat). Sains Malaysiana 39 (6): 951-955, 2010.

[10] K. H. C. Baser, T. Ozek, S. H. Beis, Constituents of the essential oil of *Ruta chalepensis* L. from Turkey. *J. Essent. Oil Res.,* 8, 413-414, 1996.

[11] F. A. Mcclure and T. M. Hwang, The flora of a Canton herb shop. Lingnan University Science Bulletin 6, 1- 32, 1934.

[12] D. C. Montgomery, *Applied Statistics and Probability for Engineers*, 3rd Edition, John Wiley & Sons, Inc., 2010.