A Comparative Physicochemical Analysis of Crude and Refined Jamun (Syzygium cumini) seed oil

Dr. Akleshwar Mathur Sr. Assistant Professor, Department of applied Sciences (Chemistry) Jodhpur Institute of Engineering and Technology, Jodhpur (Rajasthan)

Abstract- This paper deals with the comparative experimental study through extraction physicochemical characteristics of both crude and refined Jamun seed oil. The extraction was done using normal hexane. Degumming, neutralization and bleaching process were used for refining of oil, by activated clay. The specific gravity, refractive index, acid value, saponification value and iodine value were determined for both crude and refined samples, within the ASTM standard specifications. The high iodine value (94.5) for the refined oil indicates that the oil is quite unsaturated and of non-drying nature, so can be used in medicated soaps. The physicochemical properties can be improved by above refining for better applications.

I. INTRODUCTION

Syzygium cumini commonly known as jambul, black plum, Indian blackberry, jamblang or black cumin belongs to the family myrtaceae. The seed oil is yellowish brown in color and a semi viscous liquid .Jamun tree is the native of India and now spreaded in the parts of south western Asia and Eastern Africa .It is found naturally and cultivated for various products in many countries¹.

The tree fruits once in a year, which are berries and sweetish sour to taste with seed. The seeds are found effective to cure diabetes mellitus. It has antioxidant, anti-allergenic, radio protective, anti-inflammatory, neuropsycho pharmacological and anti-microbial activities.

The seeds contain 3-10 % fixed oil, containing myristic acid and oleic acid as chief fatty acids ². A wide range of phyto steroids such as beta sistosterol are also present. The fermented fruits are important raw material for wine, Brandy and distilled liquor. Jambolan vinegar is an attractive, clear purple pleasant aromatic liquid made throughout India³⁻⁷.

This paper is aimed at comparative analysis of the extracted oil with the refined oil regarding the physicochemical characterization. The following objectives are realized to achieve above aim-

- a. Extraction of Jamun seed oil from dried Jamun seeds through solvent extraction as Hexane.
- b. Refining of the crude oil.
- c. Characterization of crude Jamun oil.
- d. Comparison of both oils.

II. MATERIAL AND METHODOLOGY

A. Raw Material processing

The seeds were processed with conventional methods including collection of seeds from naturally grown trees, washing with water, drying an oven at 60°C for 7 hours and size reduction using Mortar and pestle.

B. Determination of the percentage of moisture in Jamun seeds ⁹

40 g of seeds (M) were weighed in a silica crucible (W2) and dried in an oven at 800 C for 7 hours. The weight of (crucible + seeds) sample was recorded after every 2 hours using desiccators for cooling. It was repeated to get a constant weight (W1). The moisture content (M %) was calculated

(M% = 100 (W2-W1)/M)

C. Extraction of oil and percentage of Jamun oil Extracted

300 ml of petroleum ether of 40-60 0C range was filled in a round bottom flask. 30 gram of crushed seeds was taken in a thimble and placed in the Soxhlet apparatus. It was heated at 600 C for 30 minutes. The thimble was removed from the tube, dried in oven, cooled in desiccators and the weight was taken. The same was repeated to get the maximum extract. The same process was repeated with more seeds to get good quantity of the oil.

15 g of sample seeds were taken and same procedure was repeated. After complete extraction the resulting micella was taken for solvent recovery by distillation. The oil was cooled and filled in a viol for analysis and processing. The values the % oil was determined by usual methods¹.

D. Refining of Extracted Jamun Oil 10-12

The clay sample was obtained from the local market from Jodhpur. It was grounded and mixed with water. Sand and stones were removed from it. To active the clay, 2 M HCl was added to the clay slurry and mixture was boiled for 2 hrs. at about 1000C. It was then washed to remove acid and grounded into fine powder.

E. Degumming and Neutralization

The extract was mixed with boiled water for degumming. It was stirred for 2 minutes and allowed to stand in a separating funnel. The water layer separated at upper side was discarded. The procedure was repeated with fresh water to ensure the complete removal of gummy matter.

60 g of degummed oil was heated at 800C in a borosilicate beaker and 40 ml of M/10 NaOH was added and stirred. The soap thus formed was precipitated by adding sodium chloride solution of 10% weight of the oil. This mixture was transferred to a separating funnel and allowed to stand for the separation of two layers. After one hour hot water was added again and again to complete removal of soap from the oil. The neutralized oil was taken into a beaker.

F. Bleaching of oil

50 g of oil was heated at 900C in a beaker and 7.5g of activated clay was added to it. The mixture was continuously stirred for half an hour. After that the temperature was increased to 110⁰ C for 30 minutes. The contents were kept at 700 C and filtered. The filtrate was stored in a beaker for further studies.

G. Characterization of the Extracted Jamun seed oil and refined oil

The characterization of extracted oil and refined oil were done using following methodology.

Adobe Refracto meter was used to determine the refractive index of the oils, at 300 C. Density bottle was used for specific gravity determination. The weight of 25 ml. capacity bottle was taken and it is filled with oil sample. The weight of oil and bottle was noted. The same process was repeated with water as reference standard.

ISO 3657 (1988) indicator method was applied to determine Saponification value. 2g (M) of oil was weighed in a conical flask and 25 ml. of N/10 ethanolic KOH is added to it. A reflux condenser was fixed on it and allowed to heat for 1 hour. A few drops of phenolphthalein indicator was mixed to warm mixture and titrated against M/2 HCl (N) to get the colorless solution. The volume of HCl used was noted as V_1 . The same steps was repeated with blank simultaneously also and volume was taken (V_0).

S.V. (Saponification value) was calculated by:

 $S.V. = 56.1 \text{ N } (V_0-V_1)/M.$

 $25\ ml$ of diethyl ether and $25\ ml$ of ethanol was mixed in a beaker of $250\ ml$. capacity. This mixture was added to $10\ g\ (W_0)$ of oil filled in a $250\ ml$ conical flask and a few drops of phenolphthalein indicator was mixed to it. The mixture was titrated against $M/10\ NaOH$ with continue shaking to get dark pink color at volume of NaOH V_0 . The free fatty acids were calculated as $2.82x100xV_0/W_0$. The acid value was calculated as FFA/2.

ISO 3961 (1989) method was used to determine the Iodine value. 0.4 g oil (M) was weighed into a conical flask with 20 ml. of carbon tetra chloride to dissolve it. 25 ml. of Dams reagent was added in fume chamber. The flask was swirled vigorously after inserting topper. It was placed in dark for 2.5 hour after that 20 ml of 10% aqueous KI and 120ml of water were added. It was titrating with M/10 sodium thiosulphate solution to get pale yellow solution. 1% starch indicator was added to get blue color. It was again titrated till disappearance of blue color (V₂ml). The same procedure was repeated with the blank solution (V₁ml). Then the iodine value (I.V.) is given by the expression:

I.V. =12.69C $(V_1-V_2)/M$

Redwood viscometer no.1 was used to determine the viscosity of oil sample. It was recorded as redwood second at room temperature. 50 ml of oil was allowed to flow through orifice. The time to flow of oil is noted in redwood seconds.

2g sample was taken into a clean dry beaker and 13ml of hot distilled water was added to it. And stirred slowly. It was then cooled in a cold water bath to 25°C. The pH electrode was standardized and the electrode immersed into the sample and pH value of sample was read.

All the observations were recorded and compared with reference values. Some images of current work are shown below:



Figure 1: Mature fruits and collection



Figure 2: Dried fruits and seeds



Figure 3: Crude and refined oils

III. RESULTS AND DISCUSSION

The results obtained for above mentioned analysis are shown in the table 1.

Table 1: Physico chemical properties of seeds

Sr.no.	Property of seeds	details
1	Seed morphology	Poly embryonic up to 4 embryos 3 are germinative,20-80% of fruit weight
2	Moisture content	5.65%
3	Oil content	10.0%
4	Protein content	4.99 % (NX6.25%)

Table 2: Physical properties of crude oil

Sr.	PROPERTY	CRUDE	REFINED
no.		OIL	OIL
1	Appearance	Yellowish	Pale yellow
2	Refractive	1.481	1.4673
	Index at room		
	temperature		
3	Specific gravity	0.9432	0.9333
4	Acid value (mg	1.711	0.944
	NaOH/g of oil)		
5	Saponification	180	180
	value (mg		
	KOH/g of oil)		
6	Iodine value (g	97.12	94.5
	I ₂ /100 g of oil)		
8	pН	6.22	6.41

The present content of moisture in the matured seeds was found as 5.65% which may vary from sample to sample and on the time of storage of seeds and fruits. The oil content as 10% may differ in variety, environmental and climatic conditions, also on the mode of extraction. In earlier reports it has been found in the range of 3-10%. Table 1 represents the physicochemical properties of seeds and Table 2 the physicochemical characteristics of crude and refined Jamun oil. The crude oil is yellowish in color which becomes pale yellow after treatment. The refractive index of crude oil was found as 1.481 and 1.473 for refined oil at room temperature. The minor decrease in this value may be a result of instrumental or analytical error. The specific gravity of oils was recorded as 0.9432 and 0.9333 in crude and refined oils

respectively. The acid values were found as 1.711 and 0.944 (mg NaOH/g of oil) respectively in crude and refined oils. The decrease in acid value is the result of neutralization of oil in which the free fatty acids has been neutralized by the NaOH. Saponification values (mg KOH/g of oil) were calculated as 180 for both crude and refined seed oils which are in accordance of neutralization. The Iodine values (g I₂/100 g of oil) were found as 97.12 and 94.5 respectively. It shows that there is slightly decrease in the iodine value on refining of oil. As a result of their agreement with standard categories the oils both crude and refined are classified as a non-drying oils. The acid value is more in crude oil due to presence of more free fatty acids. Decrease in pH value may be again considered as a result of neutralization. It is also concluded that more amount of alkali is required to neutralize the crude oil as compared to the refined oil whenever required. The pH of oil was found as 6.22 and 6.41 respectively. It indicates that the oil becomes less acidic due to above treatments.

IV. CONCLUSION AND SCOPE

The oil percentage in the seeds is quite satisfactory for industrial consumption. The oil is a non-drying oil so it can be used in manufacturing of soap as well as a binding material of natural drugs. The yield of seeds and their oil percentage are affected by a number of factors like environmental conditions, variety of plant, age of plant, collection timings, storage etc. Out of them many parameters can be controlled for better results. The oil produced through solvent extraction was treated with activated clay followed by degumming and neutralization. Both of the oils were analyzed for appearance, Refractive index, specific gravity, acid value, Saponification value, iodine value and pH. These values were found with small to large variations due to refining of oil.

A number of fields could be tried on the basis of present results and previous research to seek the applications of Jamun seed oil in specific manner. Since the treated oil is more clear and it is less acidic than the crude one, so this type of treatment would lead more efficient product. Further the Jamun tree is very common in almost all parts of India; the area of application would be more promissible.

In summarized form it can be concluded that Jamun seed oil should be studied further and the products should be standardized for better applications.

ACKNOWLEDGMENT

The author is thankful to Dr. M.R.K. Sherwani Retd. HOD Department of Chemistry, JNVU Jodhpur for guidance and JIET Family for support and necessary facilities.

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