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The Role of Date Seeds Oil in preparation Of Cosmetics and Food Nutrition

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Abstract

This research aimed to investigate the recycling of date seeds, which is a real practical example of the sustainable and integrated use of renewable material resources, providing innovative and sustainable solutions that can contribute to reducing environmental waste and maximizing the use of available natural resources. Highlighting the role of date seeds in supporting local industries as sustainable and safe alternative to cosmetics and pharmaceuticals. It illustrates the economical and medicinal value of date seeds waste as a natural source of active compounds. Date seeds oil was extracted, and its applications in cosmetics and nutrition were studied along with phytochemical screening tests, antioxidant and approximate analysis. Three types of extracts were prepared: methanol, aqueous, and oily. The aqueous extract was found to be rich in flavonoids (+4), tannins (+3), terpenoids (+3), saponins (+2) and absent of alkaloids (-). The approximate analysis showed moisture content (11.70%), ether extract (5.23%), fiber (75.3%), protein (8.7%), and ash (37.5%). The radical's percentage inhibition (PI) of DPPH was significantly obtained by dose $IC_{50} = 1000 \mu\text{l}$. Date seeds aqueous extract, with IC_{50} (80%) have values of 38.3, 78.9, 90.1, and 95.2 mg/mL, which contribute to its strong DPPH radical scavenging activity. Date seed oil was found to contain a high percentage of saturated and unsaturated fatty acids, phenolic compounds, and antioxidant properties, which are used in many industries, such as cosmetics (skin and hair cream, soap, eyeliner, glycerin, and coarse and smooth sandpaper), nutrition, date kernel molasses and raw material for animal feeds.

Key words: Date seeds - Proximate analysis - Cosmetics - animal feeds - Phytochemical analysis.

1. Introduction:

Date seeds are a by-product of pitted dates or for the manufacture of date paste [1]. It has a hard, coated, oblong seed with a ventral groove and a small embryo; they weigh between 4-5 g which represent 6-20% of the fruit weight, depending on maturity, variety, and grade [2]. Date seeds germinated after 74 days [3]. Date seeds wastes are used as raw materials for manufacturing household equipments, furnitures, and building materials [4]. It also used as an absorbent for water containing dyes [5]. The oil has antioxidant properties [6]. Date seeds are hard to digest, so it is necessary to treated before feeding them to animals [7]. It is preferable to soak date seeds in water for 72 hours to increase their nutritional value. [8] Grinding the seeds rise the availability of nutrients by breaking and removing the seed coat [9] Its powder beneficial for enhancing nutritional value, oxidative stress, anti-inflammatory activity, mental health, and athletic performance [10]. Date seeds are rich in antioxidants (580–929 mL Trolox equivalents/g date seeds and phenolic compounds (21.0–62.0 mg gallic acid equivalents (GAE)/100 g date seeds [11-12]. Date seeds are still regarded as a by-product and typically utilized as animal feed, despite their high fibers content. Dairy cows were the first to be fed by date seeds in animal feed [13]. As well as in fisheries nutrition [14] Goats [15] Broilers [16]. It was shown that the use of date seeds in animal feeds improved the growth and raised the levels of testosterone [17] and estrogens [18].

Material and Methods

2. Materials plant

Date pits were obtained from local roasters and herbal markets in Sharurah, cleaned of impurities, washed with water, dried in the shade, and then oven-roasted at 150°C.

2.1. Palm Plant Classification

Date palms are classified as *Phoenix dactylifera* (*Viridiplantae*), belonging to the *Arecaceae* family, which include only one genus and one species in Saudi Arabia [19].

2.2. Preparation of oil and methanol extracts from date pits :

The course (500 g) of fully crushed date pits was weighed, repackaged, and extracted by using methanol and petroleum ether (1:2) in a Soxhlet Grant Instruments Ltd. The methanol and oil extracts were filtered and evaporated under reduced pressure using a rota-vap. The extracts were weighed, recorded, and then refrigerated until ready for use. For the aqueous extract, 100 g of the sample is soaked in 500 ml of hot distilled water for 4 hours, then filtered using filter paper (No. 4). Then extracts kept in the refrigerator for 48 hours, then placed in an oven at 350°C until completely dry. The remainder is weighed and the extract percentage is determined according to the equation below [20].

$$\text{Yelid (\%)} = \frac{\text{weight of sample before extraction}}{\text{weight of sample after extraction}} \times 100$$

2.3. Chemical Test:

2.3.1. Fehling's A+B Reagent Test:

Fehling's solution (Rochelle salt) consists of a blue aqueous solution of copper (II) sulfate (A) and a colorless solution of aqueous sodium and potassium tartrate (B).

2.3.1.1. Procedure:

Add 1 ml of the aqueous extract in a glass test tube with 1 ml of Fehling's solution (A+B). Shake it lightly to mix the mixture, then place it in a water bath for 3-4 minutes.

2.3.2. Extraction of Polysaccharides and Hemicellulose from Date Seeds

Polysaccharides (DSP) and hemicellulose (DSH) extraction has been separated according to methods [21-22]. As shown in fig (1).

30g of seed powder was de-oiling using 90% methanol and kept for 24 hours. Dilution of 10 times was performed on the defatted date seed powder, followed by incubation at 100°C in a thermostatic water bath for 120 min and filtration. The DSP and DSH fractions were recovered by supernatant and residue respectively. In order to precipitate the DSP, the supernatant was first concentrated ten times using a rotary evaporator under vacuum at 50°C. Four volumes of 90% ethanol were then added, and the mixture was then incubated for 24 hours at 4°C. Centrifugation was done for 15 minutes at 5000 rpm to separate the precipitated DSP from the supernatant. The precipitate (DSP) was then re-suspended in deionized water, and the supernatant was disposed off. Minerals and low molecular weight molecules (cut off = 1 kDa) were discarded during washing from the extract. Drying step and freezing. Powdered DSP was obtained following a freeze. The remainder is weighed, and the recovery yield (% (w/w)) is calculated according to equation below:

$$\text{DSP recovery yield (\%)} = \frac{\text{Lyophilized DSP weight (g)}}{\text{Date seed powder weight (g)}} \times 100$$

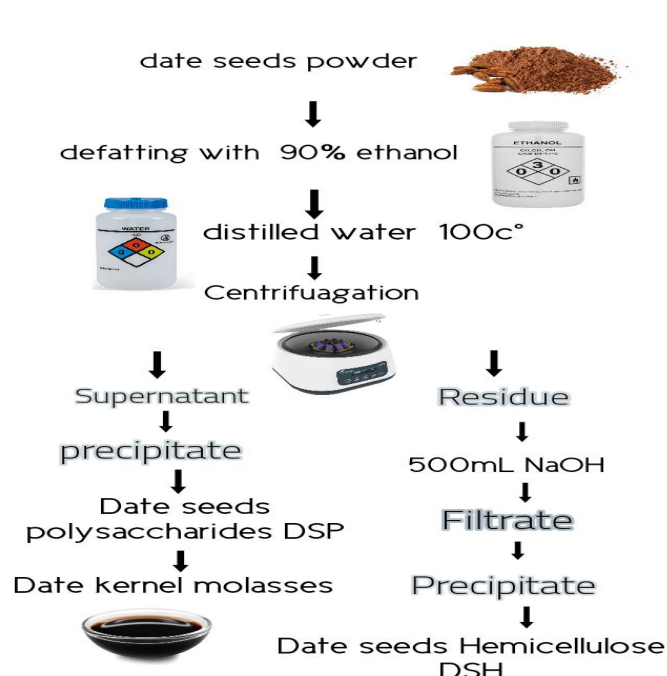


Fig 1: Diagram of extraction of polysaccharides and hemicellulose from date seeds

2.3.3. Copper acetate test:

Add 2 ml of vegetable oil in test tube as control and 2 ml of date pits oil in the other tube. Then, add 3 ml of petroleum ether and shake, followed by 3 ml of copper acetate in both tubes. All layers will appear blue, indicating the presence of saturated and unsaturated fatty acids. In the control tube, two layers were separated; the upper layer, with a blue color, indicates a negative result [23].

2.4. Phytochemical Screening Test

2.4.1. Tannin:

1 g of date seed powder was dissolved in 10 ml of hot saline and divided into two test tubes. 2-3 drops of ferric chloride were added to the first tube, followed by 2-3 drops of Siegelman's gelatin salts indicator to the second tube. When a blue-black color appeared in the test tube this indicate the presence of tannin [24].

2.4.2. Terpenoids test:

1 g of date seed powder was dissolved in 10 ml of chloroform, and then 10 ml of acetic anhydride and 3 drops of sulfuric acid were added to a test tube. At the point of contact between the two liquids, a gradient of green, red, and blue colors appeared, indicating the presence of terpenoids [25].

2.4.3. Flavonoids: test

1 g of date seed powder was dissolved in 30 ml of 80% ethanol, then filtered and 1 ml of potassium hydroxide solution was added to a test tube. The appearance of a dark yellow color indicates the presence of flavonoid compounds (flavonoids, flavonols, and flavonones) [26].

2.4.4. Saponin test:

Add 3 ml of the aqueous extract to a test tube, then add 10 ml of distilled water, and shake the tube vigorously for approximately 30 seconds. A foam will appear and lasts for at least 15 minutes, this indicate presence of saponins [25-26].

2.4.5. Alkaloid test:

Add 5 ml of the aqueous extract to a test tube, then add 1 ml of diluted picric acid. A yellow precipitate is observed, indicating the presence of alkaloids [25].

2.5. Approximate Analysis:**2.5.1. Moisture content determination:**

Moisture is determined according to method [27]. The sample is dried in an electric oven at 100-150°C for 18 hours

2.5.1.1. Procedure:

- Weigh the crucible (WC).
- Weigh 2 grams of sample (WS)
- Record the weight of the crucible and sample.
- Place the sample in an oven at 150°C for 18 hours.
-

$$\text{Moisture (\%)} = \frac{(WC + WS)_b - (WC + WS)_a}{WS} \times 100$$

2.5.2. Ash content determination:

The percentage of raw ash present in the sample after combustion in a furnace at 550-600°C for 3 hours. is determined according to method [28].

2.5.2.1. Procedure:

- Weigh the empty crucible (WC).
- Weigh 2 g of the sample (W S)
- Place the crucible and its contents in an oven for 3 hours at 550-600°C.
- Allow the crucible to cool at room temperature.
- Record the crucible weight and ash content.

$$\text{Ash}(\%) = \frac{\text{Weight of the crucible and ash} - \text{WC}}{\text{Weight of the sample}} \times 100$$

2.5.3. Estimating the percentage of crude fat.

Estimating the percentage of crude fat was done according to method [27-28], where the percentage of fat from the sample powder is determined by extraction using petroleum ether at a ratio of (1:1).

2.5.3.1. Procedure:

- Weigh an empty beaker (W1).
- Place a piece of cotton in the bottom of the extraction thimble.
- Weigh 2 g of sample and place it in the extraction apparatus (W2).
- Add 100 ml of petroleum ether into a 150 ml round bottom beaker.
- Assemble the extraction unit over an electric heating mantle.
- Heat the solvent in the beaker to boiling point. Then, adjust the heat source to 80-90°C, allowing the solvent to drip from the condenser into the sample chamber.
- The extraction continues for 6 hours.
- The extraction unit is removed from the heat source and the beaker is placed on top of it until the remaining solvent has completely evaporated (S).
- The beaker is left to cool, then its contents are weighed.
-

$$\text{Fat} (\%) = \frac{(w2 - w1)}{S} \times 100$$

2.6. Anti-oxidant activity of date seeds

DPPH free radical scavenging assay was used to assess the antioxidant activity of the examined extracts [29]. Radical scavenging action of (2,2-diphenyl-1-picrylhydrazyl -DPPH).The radical methanol solution (0.004% w/v) was made and kept in the dark at 10°C. 500µl of each extracted solution and 500µl of DPPH solution were combined in the testing tube. Instantaneous absorbance measurements were taken using an Agilent Cary-100 UV-visible spectrophotometer (Germany). Data was continually captured at one-minute intervals to determine the decline in absorbance at 515 nm intervals for 16 minutes, or until the absorbance stabilized. Measurements of absorbance were made to ascorbic acid as a concern molecule (control). The following formula [30] was used to determine the DPPH radical's percentage inhibition (PI):

$$\text{PI} = \frac{(\text{AC} - \text{AT})}{\text{AC}} \times 100$$

Where AC= Absorbance of the control at t = 0 min and AT= absorbance of the sample + DPPH at t = 16 min. The IC₅₀ value of each antioxidant assay was calculated and expressed as µl/mL

2.7. Preparing cosmetics from date seeds oil

2.7. 1. Soap Preparation.

- Soap is prepared according to method [31].
- Weigh 6 ml of oil in a beaker, then add 50 ml of 80% ethanol to dissolve the oil.
- 30 ml of sodium hydroxide solution NaOH.
- Heat the mixture in a water bath to 80-85°C without boiling. Stir for 20 minutes, then test for complete saponification.
- Prepare a solution consisting of 40 g of sodium chloride dissolved in 200 ml of water. Place it in an ice bath and divide it in half.
- After complete saponification, allow the soap to dry and note the resulting weight.

2.7.2. Transesterification process for glycerol production:

Add 10 ml of date seed oil in a round-bottom flask, then weigh and thoroughly dissolve in 20 ml of ethanol with 1 g of the catalyst (date pits) for 3 hours at 80°C. Then, the solution was placed in a separating funnel, and the product was left for 24 hours for the separation process [32], as shown in fig (2).

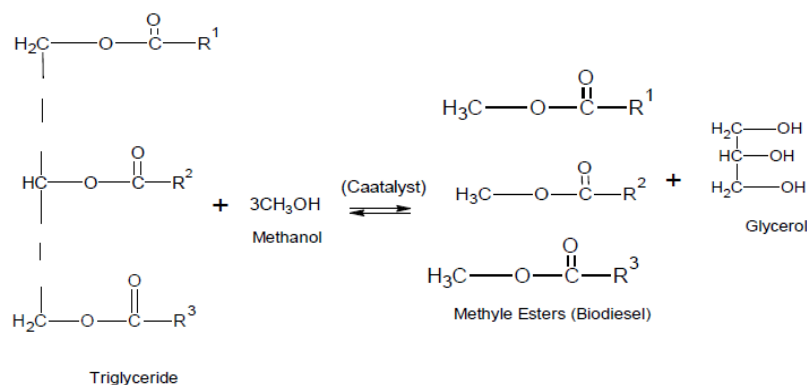


Fig 2: diagram of Trans-esterification process for glycerol production

2.7.3. Cream manufacturing

Add 1 g of polyethylene glycol to 10 ml of date seed oil and put the mixture in a water bath at 70°C to form

a brown cream with a soft texture. Polyethylene glycol, which is characterized by being non-toxic, colorless, inert, odorless, non-volatile, and viscous at normal room temperature [33].

2.7.4. Raw material for animal nutrition

Common preparation methods included grinding, soaking, and chemical or biological treatment.

- Grinding: Breaks hard shell, used crushing and hummer mill to assess nutrients.
- Soaking : 72 hrs softens seeds, aids digestibility, minimal impact on intake.
- Chemical treatment (NaOH): Improves fiber digestibility effectively.
- Biological treatment it can be incorporated in to feed mixtures to improve their stability and shelf life, also improved feed preservation and reduced spoilage [34].

3. Result and Discussion

Date seeds are shown in fig (3&4). Its oil is characterized by a moderate viscosity ratio, a white color, and a liquid state at room temperature [35].



Fig 3: Date seeds

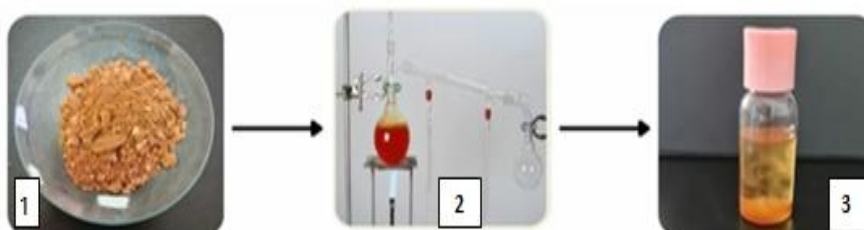


Fig 4: Date seed powder (1) Extraction device (2) Oil extract (3).

3.1. PH test

Refer to figure (5), shows that the pH of date seed oil is ≥ 7 .

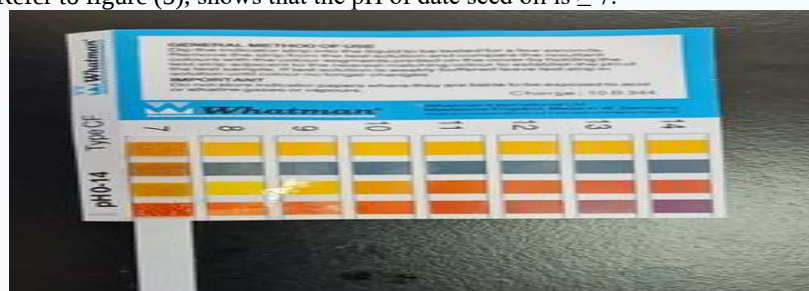


Fig 5: pH ≥ 7

3.2. Protein Test:

Figure (6) shows the Biuret test in date seed oil. A dark blue color appearing in test tube (B) indicates the presence of protein.



Fig 6: biuret's test

3.3. Copper acetate test:

Refer to figure (7). In test tube no (B) all layers are colored into a dark blue, indicating the presence of saturated and unsaturated fatty acids, when compared to the base or control tube no. (A), we notice the formation of a blue layer on the surface.

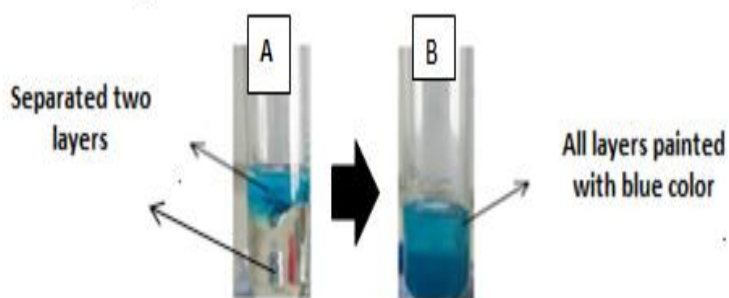


Fig 7: Copper acetate test

3.4. Fehling's A+B Test

Figure (8) appearance of a reddish-brown color when performing the Fehling's test with aqueous extract of date seeds which indicating the presence of sugars (mono-di-polysaccharides).



Fig 8: Fehling test

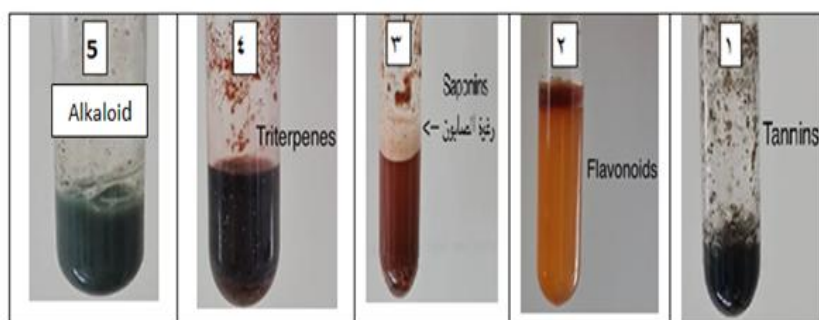
3.5. Phytochemical Screening Test

Appearance of a blue-black color in the test tube, indicating the presence of tannin (+3), while in Figure (2), the yellow color indicates the presence of flavonoid compounds (+4). Figure (3) shows the formation of foam that lasted for at least 15 minutes, indicating the presence of saponin (+2). As for figure (4), a blue-green color appears at the bottom of the tube, indicating the presence of terpenoids (+3). We note in figure (5) that the yellow precipitate does not form, indicating the absence of alkaloids (-), as table (1) & figure (9).

Table no (1): Phytochemical Screening Test

Test	Tannin	Flavonoid	Saponin	Terpenoids	Alkaloids
Methanolic extracts	+++	+++	+++	+++	-
	a blue-black color	Yellow colour	formation of foam that lasted for at least 15 minutes	blue-green color	NP

Key: (+) Positive result, (-) Not Present



3.6. Proximate analysis of date seeds extract and animal nutrition

Date seeds can be used as a component of animal feeds, but require processing to improve digestibility and nutrient availability for livestock. Table (2) display various date pits compounds of valuable source such as: moisture content (11.70), ash (37.5), ether extract (5.23), fiber (75.3) and protein (8.7 g/100 g) respectively, having a potent antioxidant activity due to their composition with flavonoids and other components.

Table no (2): Approximate analysis

Test	Moisture (%)	Ash (%)	Ether extract (%)	Fiber (%)	Protein (%)
Petroleum ether extract	11.70	37.5	5.23	75.3	8.7

3.7. Anti-Oxidant activity

DPPH radical scavenging activity of date pits indicate in table no (3). The radical's percentage inhibition (PI) of DPPH was significantly obtained by dose sample with $IC_{50} = \mu\text{l} / 1000 \mu\text{l}$. date aqueous extract, with IC_{50} (80%) values of 38.3, 78.9, 90.1 and 95.2 mg/mL, which contribute to its strong DPPH radical scavenging activity compared to the control (Ascorbic acid).

Table (3): DPPH scavenging activity of date seeds extracts.

Sample Doses	DPPH scavenging activity (%) / Sample conc ($\mu\text{l} / \text{mL}$)					IC_{50}
	12.5	25	50	100	200	
Aqueous extract	ND	38.3	78.9	90.1	95.2	80.3
Ascorbic acid	18.77	57.3	91.28	100	100	20.6
^a Ascorbic acid was used as standard Anti-oxidant agent at 100 $\mu\text{g}/\text{mL}$, ^b ND: not determined						

3.8. XRD analysis

Mineral salts (Na, Mg, P, K and Ca) have been measured in heat treated date seeds at 800°C as fig (10). It was demonstrated that it had elevated calcium levels 71.02% and moderate level of sodium 32.39%, magnesium 26.04 %, potassium 1.47% and phosphor 0.32% [36].

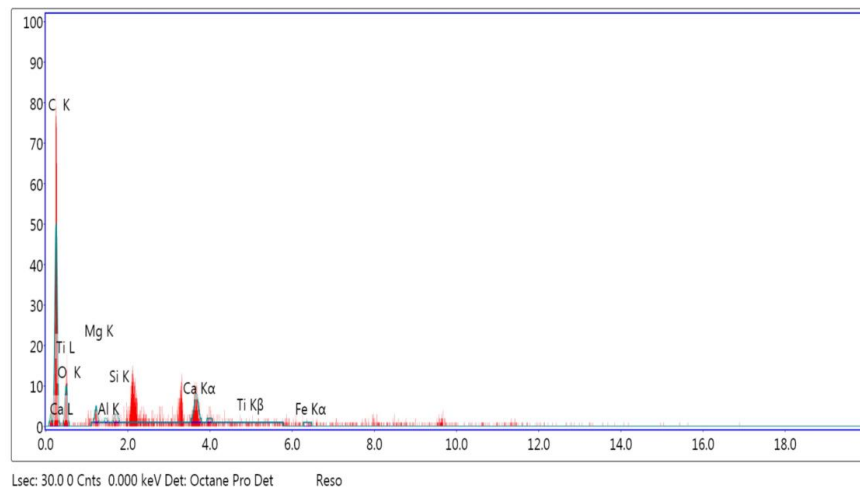


Fig 10: Measurement of mineral date pit

3.9. Preparing cosmetics from date seeds extract

The cosmetics industry in Saudi Arabia has undergone a major transformation in terms of regulatory aspects and quality control measures. The implementation of the 2020 regulations has reshaped the Saudi cosmetics market, bringing it closer to international standards while addressing local challenges. Figure (11) shows the characteristics of the produced soap, is thick lather with smooth texture.

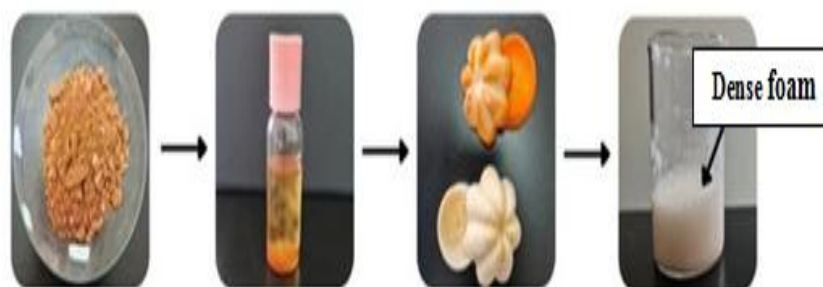


Fig 11: Preparation of solid soap and the appearance of dense foam

Figure (12) Glycerol production: when using heat-treated date seeds catalyst at 900 °C, two layers were formed, the lower layer is glycerol, which is characterized by white color and heavy consistency.

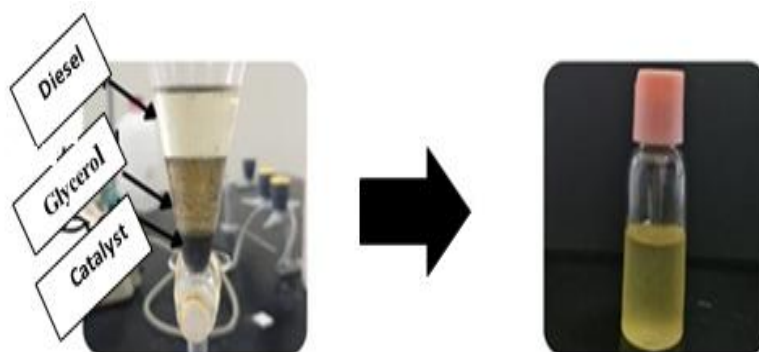


Fig 12: Glycerol production

Figure (13) shows the black powder of kohl, which has formed small, smooth black crystals through the process of sublimation, which consider being a good method for purifying compounds.



Fig 13: Eye liner (Kohl) manufacturing:

Figure (14) shows kinds of sandpaper (A) which has a rough texture due to its inclusion of date pits, whereas smooth sandpaper (B) has sweet-smelling, soft-textured.



Fig 14: Coarse (A) & smooth (B) sandpaper

Figure (15) display the formation of a light brown cream with a smooth texture and a heavy consistency.

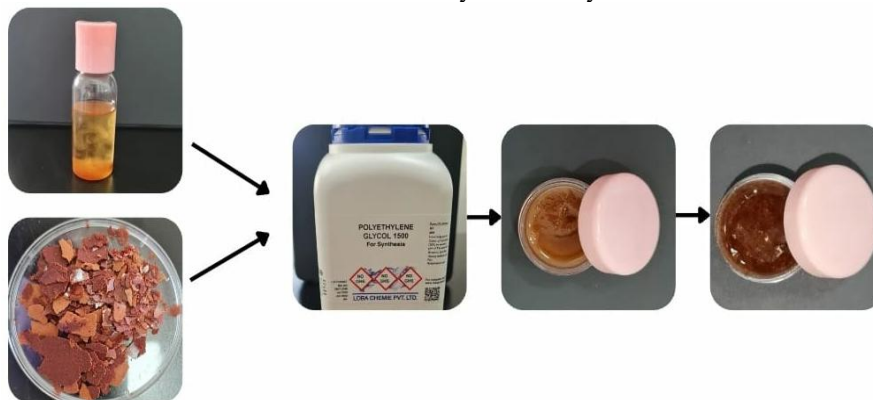


Fig 15: A light brown cream process.

Figure (16) show the process of date seeds as animal feeds, after date seeds are ground, soaked, and chemically treated with sodium hydroxide, they are baked in the form of spherical granules, then left for a week to dry. Final products are free of fungi and rich in fiber and sugar. It is presented to animals as food feed.



Fig 16: Animal feeds process

Diagram (1) shows the process of conversion of polysaccharides, which are placed in low heat with continuous stirring, it turns into date kernel molasses with a dark brown color and thick texture.

Conclusion

It has been proved that date seed extracts can be used as an important resource in traditional medicine and cosmetics, instead of being discarded as agricultural wastes. The importance of date seed extracts is due to their physical and chemical properties, in addition to the various natural products they contain, such as mono terpenoids, aromatic volatile oils, flavonoids, and saturated and

unsaturated fatty acids. The antioxidants in it play an important role in protecting skin cells and improving skin elasticity and minimize the appearance of wrinkles. The oil has been used in many fields, including cosmetics, perfumes, food, and medicine. We hope to conduct further research into date pits and their extracts for nanotechnology applications in the future.

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