

Original Research Article

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Study on Physico-chemical Properties of Oil and Powder of Date Palm Seeds (*Phoenix dactylifera*)

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ABSTRACT

Keywords

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Date palm seeds (*Phoenix dactylifera*) are discarded as waste after utilization of date flesh by the industries. But the oil from date seed is reported to be rich in unsaturated fatty acids, phenols and tocopherols. Whereas, the date seed powder (DSP) contains high protein and fibre content. Among all the treatment (Control, Soaking, Soaking + Roasting, Roasting) control sample showed highest oil yield (8.2%) was observed. The total fat content in the DSP was 10.7%, Protein (5.5 g/100g) and Total dietary fiber (57.24%), with 52.7% of it was insoluble dietary fiber. The date seed oil is rich in unsaturated fatty acids with oleic acid as the predominant one (51.456%) followed by palmitic acid (12.831%), Myristic acid (10.447%), Lauric acid (10.363%), Linoleic acid (7.20%) and Stearic acid (6.56%). The percent of FFA (Free Fatty Acids) was (0.9 %). The low peroxide value of 1.243 meq O₂/kg indicates that date seed oil is fresh and is less susceptible to autoxidation. The iodine value is 73.60g Iodine /100g. The high saponification value of 208.33 mg KOH/ g of oil indicates very high content of low molecular weight triacylglycerol, which is an desirable property to use such oil as bio diesel. The best oil yeild was satisfactory at 2 h extraction time and 2000 microns mesh size by using soxhelt extractor and petroleum ether as solvent.

Introduction

Date seeds, also called stones or pits, form part of the integral date fruit, which is composed of a fleshy pericarp and seed that constitutes between 10% and 18% of the date fruit's weight, depending on the variety and quality (Hussein *et al.*, 1998); thus, approximately 12,25,000 tons of date seeds are produced annually (FAO, 2016). As it is also known that date seeds contain valuable bioactive compounds, utilization of this by-product is highly desirable for the date industry. The seeds are often used as animal

feed-stuff (Ahmed *et al.*, 1995), propagation of date palms, and other industrial applications. The palm pit oil contains a certain percentage of pale yellowish-green oil with pleasant odor (Shahidi, 2005).

The palm kernel oil is used for several applications like a liniment for indolent tumors (Graham *et al.*, 2000), and showed antimicrobial effects against some microorganisms like *Escherichia coli*, hemolytic *Streptococci*, *Aspergillus fumigatus*

and *Staphylococcus aureus* (Barmak, 2011). It is a rich source of some minerals, especially iron content, total sugars and crude fats (Al-Shahib and Marshall, 2003), large quantities of fiber, and possibly resistant starch (Ekwenye and Ijeomah, 2005).

As the protein and fibre sources which are available in the present era are more expensive. This has led to the investigation of alternative nutritive sources for food use which can be used to either partially or fully replace more expensive ones. There is also an advantage in using non-utilized by products that were considered to be waste to recover useful functional nutrients. One such waste product from food industry is Date Seed. As Date Seed contains considerable amounts of proteins, dietary fibre and oil. Keeping this in view present research was under taken with the objective of extracting the oil from date seed and studying the oil characteristics and fatty acid profile along with evaluating physico-chemical properties of defatted date seed powder.

Materials and Methods

Raw material and processing

Semi dry dates were procured from local market of Bapatla, Guntur District, Andhra Pradesh, India. Date seeds are manually removed pulverized to a mesh size of 2000 microns. After manually separating the date seeds they are subjected to different treatments (Control, Soaking, Soaking + Roasting and Roasting). Then the pulverized date seeds were subjected for oil extraction in Soxhelt Apparatus.

Physico chemical analysis of date seed oil and defatted date seed powder

The Date palm seed oil and powder extracted were evaluated for their physico-chemical

analysis namely, moisture content, carbohydrates, reducing sugars, protein, fat, Phenolic compounds, crude fiber, ash content, tocopherol, peroxide value, iodine value, acid value, saponification value and unsaponification value.

Moisture content

The moisture content of date palm seed powder was determined by air oven drying method by placing about 2 g of sample for 24 h in an air oven maintained at 103 ± 1 °C (AOAC, 1980).

Total carbohydrate

The Total Carbohydrate content can be measured by Phenol Sulphuric Acid method

Absorbance corresponds to 0.1 mL of the test = x mg of glucose

100 mL of the sample solution contains = $x/0.1 * 100$ mg of glucose

Fat content by soxhlet method

The fat content of the samples is estimated by Soxhlet method (AOAC, 1980)

$$\text{Fat content (\%)} = \frac{(\text{Final weight of beaker} - \text{Weight of empty beaker}) * 100}{\text{Weight of sample}}$$

Protein content

The protein content is measured by using Micro Kjeldahl method

$$\text{Nitrogen g/kg} = \frac{(\text{ml HCl} - \text{ml Blank}) * \text{Normality} * 14.01}{\text{Weight of the sample}}$$

$$\text{Protein} = \text{Nitrogen} * 6.25$$

Crude fiber

$$\% \text{ Crude fibre} = \frac{(w_2-w_1) - (w_3-w_1) * 100}{\text{Weight of the sample}}$$

Ash content

Ash content of the sample was done in muffle furnace

Iodine value

The Iodine value is a measure of the degree of unsaturation in oil. It is constant for a particular oil.

Iodine value is a useful parameter in studying oxidative rancidity of oils since higher the unsaturation the greater the possibility of oils to go rancid.

Iodine value/ number was defined as the 'g' of iodine absorbed by 100 g of oil.

$$\text{Iodine number} = \frac{12.69 (B-S) N}{\text{g Sample}}$$

Where,

B = mL thiosulphate for blank, S =mL thiosulphate for sample

N = Normality of thiosulphate solution

Acid value

A small quantity of free fatty acids is usually present in oils along the triglycerides. Free fatty acid content is known as acid number/ acid value. It increases during storage. Keeping quality of oil, therefore, release upon the free fatty acid content. Acid number was defined as mg of KOH required to neutralize the free fatty acids present in 1 g of sample.

$$\text{Acid value} = \frac{\text{Titre value X Normality of KOH X 56.1}}{\text{Weight of the sample (g)}}$$

Saponification value

Saponification is the process by which the fatty acids in the glycerides of the oil are hydrolysed by an alkali.

Saponification value is the amount (mg) of alkali required to saponify a definite quantity (1 g) of oil. This value is useful for comparative study of the fatty acid chain length in oils.

$$\text{Saponification value} = \frac{28.05 \text{ X (Titre value of blank – Titre value of sample)}}{\text{Weight of the sample (g)}}$$

Peroxide value

Peroxide value is a measure of the peroxides contained in the oil. The peroxides present are determined by titration against thiosulphate in the presence of KI. Starch is used as indicator.

$$\text{Peroxide value} = \frac{S \text{ X N X } 100}{\text{g sample}}$$

Where, S = mL sodium thiosulphate (Test - blank) and N = Normality of sodium thiosulphate

Phenols

Phenols react with phosphomolybdic acid in Folin – Ciocalteu reagent in alkaline medium and produce blue colored complex (molybdenum blue).

From the standard curve, the concentration of phenols in the test sample is expressed as mg phenols/100g material.

Tocopherols

Tocopherols can be estimated after Emmerie – Engel reaction. This is based on the reduction of ferric to ferrous ion, by tocopherols, which then forms a red color with 2,2',dipyridyl reagent. Tocopherol is first extracted into xylene and read at 460 nm; a correction is made after adding ferric chloride and read at 520 nm.

Amount of tocopherol in $\mu\text{g/g}$ of tissues = $\text{OD at } 520 \text{ nm} - \text{OD at } 460 \text{ nm} * 0.29 * 15 * \text{Total volume of homogenate/Reading of standard at } 520\text{nm} * \text{volume used} * \text{Weight of tissue}$

Unsaponification

Unsaponifiable matter represents substances which are insoluble or are incapable of forming soaps with alkali. In edible oils, it is present upto 1-2%.

The crystals of unsaponifiable matter are observed for the presence of this steroid to know the animal or plant origin of the oil. In the method, the oil is saponified with alcoholic KOH and then extracted with petroleum ether. The extract is washed with alcohol and water, evaporated and weighed. Unsaponifiable matter is represented as difference between the weight of the residue and the fatty acids content of it (determined by NaOH titration in alcoholic medium).

Amount of fatty acids in extract = B

B as oleic acid = $0.282 * V * N$

Where,

V = Titration reading,

N = Normality of NaOH

Percentage of unsaponifiable matter = $100(W_1 - B)/W_2$

Where,

W_2 = wt. of sample

W_1 = wt. of residue.

Results and Discussion

Oil extraction

Oil from Date palm seed was extracted by using different combinations of soaking and heating procedures. Among all the treatments control sample showed highest oil yield of 8.2% followed by T₃ -Roasting (7.9%) and T₁- Soaking (7.8%) and least oil (7.2%) yield was obtained in T₂ = Soaking + Roasting (7.2%) sample.

Effect of particle size

It has been noticed that the decrease in particle size leads to increase of oil yield. This manner is expected because of the increased surface area of grounded seed. For this reason, the contacted area between seed and solvent increased, and the mass transfer of oil from the solid phase to the liquid phase increased accordingly. Also, the time needed for the solvent to diffuse inside the small particle seed is lower than large particle. Highest oil yield (8.2%) was observed in seed powder having mesh size of 2000 microns.

Physico chemical properties of date seed powder

Initial Moisture Content of the DSP was 4.8% and after extraction of oil from the DSP, DDSP recorded a moisture content of 6.5% this may be due to the presence of traces of solvent in DDSP.

Fat content in the DSP was 10.7%, after oil extraction by soxhlet apparatus using petroleum ether as solvent the oil residue present in DDSP was 2.55%. On the other

hand, protein also had been detected to be present in date seed in considerable amount of 5.5 g/100g in DSP and 5.32 g/100g in DDS. The total dietary fiber found in date seed was 57.24%, with 52.7% of it was insoluble dietary fiber namely as hemicelluloses, cellulose and lignin (Al-Farsi and Lee, 2008b). The carbohydrate present in DSP is 78.3% and that of DDS is found to be 76.8%, this could be due to the presence of sugars like sucrose and invert sugar. The total sugars in DSP is 4.50 g/100g which accounts to be 2.46 g/100 g of reducing sugars and 1.98 g/100 g of non-reducing sugars whereas the total sugars in DDS is 4.01 g/100 g of which the reducing sugars accounts to be 2.23 g/100 g and the non-reducing sugars to be 1.78 g/100 g. This could be due to the presence of appreciable amount of free aldehydes and ketone groups present in the date seed powder.

Chemical properties of date seed oil

The percent of FFA (Free Fatty Acids) (0.9%) and its value of 1.83mg KOH/g is very low due to its high content of unsaturated fatty acids. Low FFA indicates that the oils contain a small amount of free fatty acids, and this could be perhaps due to the small exposure of the seeds to the air during the maturity of the fruits of the dates. The low free fatty acid

(FFA) content of the oil shows that it is edible and could have a long shelf life when compared with many other edible oils.

The saponification value gives an indication on the nature of the fatty acids, which contains the fat and that depends on the average molecular weight of these fatty acids. The high saponification value of 208.33 mg KOH/ g of oil indicates very high content of low molecular weight triacylglycerol, which is an desirable property to use such oil as bio diesel. The high saponification value of date seeds oil indicates that the fatty acids present in the oil have high number of carbon atoms. This means that date seeds oil, after hydrogenation, could be substituted for some conventional oils.

The low peroxide value of 1.243 meqO₂/kg indicates that seed oil is fresh and is less susceptible to autoxidation. These variations can arise from different factors such as the degree of unsaturation of the fatty acids present in the particular oil, storage, exposure to light, and the content of metals or other compounds that may catalyze the oxidation processes (Choe and Min, 2006). In general, the date seeds oil can be considered as safe for human consumption, because of its low peroxide value that is less than 30 meq peroxide/kg (Gotoh and Wada, 2006).

Table.1 Fatty acid profile of date seed oil

Retention time (min)	Identified compounds	Saturation	Weight (%)
1.773	Capric acid	C10:0	0.713
1.9	Lauric acid	C12:0	10.363
2.13	Tridecanoic acid	C13:0	0.103
2.311	Myristic acid	C14:0	10.447
2.557	Pentadecenoic acid	C15:0	0.068
2.883	Palmitic acid	C16:0	12.831
3.338	Heptadecanoic acid	C17:0	0.14
3.922	Oleic acid	C18:1	51.456
4.613	Stearate acid	C18:0	5.56
4.766	Linoleic acid	C18:2	7.20

Iodine value is a measure of the unsaturation of fats and oils and is expressed in terms of the number of gram of iodine absorbed per 100 gram sample. The iodine values of (73.60g Iodine /100g) indicate that this oil is non-drying, highly unsaturated oil. Since the iodine value of date seed oil is lower than 100 it could only be classified as a non-drying oil.

Among 10 fatty acids that were detected Oleic acid was the predominant one (51.456%) followed by palmitic acid (12.831%), Myristic acid (10.447%), Lauric acid (10.363%), Linoleic acid (7.20%) and Stearic acid (6.56%) which composed together more than 90% of the total oil.

Generally, oils with high oleic fatty acid contents showed good flavour and frying stability. Oleic fatty acid is beneficial to health due to its low saturation level, minimal *trans*-isomer level and its potential to reduce LDL cholesterol in the blood as well as high oxidative stability.

The vitamin E content in date palm seed was reported as 6.946 µg/g which is a fat soluble vitamin. Medically, vitamin E is a potent antioxidant that protects the body against oxidation reactions (radicals) that damage membranes cholesterol transporting lipoproteins. Other medical use cover activity as screening reagent protecting against skin damage and aging by UV radiation, inhibits growth of cancer cells and a protector against atherosclerosis by lowering cholesterol level.

Biodiesel is assumed as an alternative to conventional diesel. Normally biodiesel is made from vegetable oil, which has few advantages than the diesel in terms of chemical and physical properties such as being biodegradable, non-toxic, its renewability, better gas release, and it yields particles, smoke and carbon monoxide at lower level. In biodiesel production, the oil

will react with alcohol such as methanol to produce methyl ester, which is known as biodiesel (Van Gerpen, 2005).

Many oils shows that with high free fatty acid content will result in more catalyst needed (potassium hydroxide (KOH) or sodium hydroxide (NaOH) in order to balance the acidity of the oil. This would become as an advantage for the date seed oil as the free fatty acid content in this oil is very low (0.9%). The high saponification value of 208.33 mg KOH/ g of oil indicates very high content of low molecular weight triacylglycerol, which is an desirable property to use such oil as bio diesel.

Date seed oils had a low refractive index indicating that they may contain fatty acids with medium-short hydrocarbon chains. Viscosity of date seed oil is one of the lowest among most vegetable oils but similar to that of olive oil. This is also an indication to use date seed oil as biodiesel.

The oil extracted from palm seeds is very much similar to other bio oils in chemical composition and basic fuel properties. The best oil yield was satisfactory at 2 h extraction time and 2000 microns mesh size by using Soxhlet extractor and petroleum ether as solvent.

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