



# Optimization of oil extraction from waste “Date pits” for biodiesel production



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## ABSTRACT

Biodiesel produced from non-edible feedstocks is increasingly attractive alternative to both fossil diesels and renewable fuels derived from food crops. Date pits are one such lipid containing feedstock, and are widely available in Oman as a waste stream. This study analyses the effects of soxhlet process parameters (temperature, solvent to seed ratio and time) on the extraction of oils from waste Date pits and the subsequent production of biodiesel from it. The highest yield of oil extracted from the Date pits was 16.5 wt% obtained at a temperature of 70 °C, solvent to seed ratio of 4:1 and extraction duration of 7 h. Gas Chromatography analysis showed that Date pits oil consisted of 54.85% unsaturated fatty acids (UFA). Transesterification of the oil extracted was undertaken at 65 °C, a methanol to oil ratio of 6:1 and a reaction time of 1 h for biodiesel production. Biodiesel produced from the Date pits oil was found to have a cetane number of 58.23, density 870 of kg m<sup>-3</sup>, cloud point of 4 °C, pour point of -1 °C, CFPP of -0.5 °C and kinematic viscosity of 3.97 mm<sup>2</sup> s<sup>-1</sup> (40 °C). In general, Date pit oil appears to be a potential alternative feedstock for biodiesel production.

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## 1. Introduction

As the industrialization of economies across the world continues to advance, and with a rapid increase in the world population, the demand and usage of fossil based fuel has increased to enormous levels [1,2]. The current level of reliance on fossil fuel energies is unrealistic as man continues seek to secure adequate supplies of energy sources [3], and the increasing reliance on fossil fuels to meet this increasing energy demand will also result in an increase in the emissions of greenhouse gases into the atmosphere, leading to severe environmental problems [4,5]. Statistics suggest that if the consumption of fossil fuel follows the same current energy consumption trend, there will be an increase of 60% in carbon dioxide emissions within the next 20-years [6]. It would not be wrong to say that if global fossil fuel use for energy production continues at the present speed, a time will come that this planet would not be a suitable place for living [7,8]. For all these reasons, much effort is currently invested in the development of alternative energy sources [9].

The transition from fossil fuels to alternative energy systems that utilize renewable energy sources has now become indispensable for sustainable environmental and economic growth [10–12]. This transition presents a challenge not only for researchers and technologists but also in considering economic and environmental impacts [13]. In addition, it is also important for the harmonious coexistence of human and for a sustainable development [14,15]. Increasing global energy demand can be met by considering various energy sources, with the global energy mix undergoing a transition from an energy portfolio dominated by fossil fuels to one that includes a range of alternative fuels [16,17]. It has been suggested that biomass-derived fuels will play a vital role in fulfilling the world's future energy demands [18]. Relative to the direct use of biomass as a fuel, liquid biofuels derived from biomass have several advantages such as high energy density, easier transportation and reduced storage volume for equivalent energy content [19,20]. In order to make biodiesel industrially viable and avoid food vs fuel controversy non-edible source are preferred. Various non-edible feedstocks have been studied for use in the production of biodiesel, with several of these found to have a high FFA content, for example jatropha seed oil (FFA 15%) [21], mango seed oil (FFA 6.6%) [22] and neem oil (FFA 12.02%) [23]. However, oils extracted from Date pits oil have been reported to have a FFA 0.59%, suggesting that, unlike

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oils from some other non-edible feedstocks, it could be used for biodiesel production without any pre-treatment to remove or transesterify the FFA content [24].

Date palm, a tropical and subtropical tree which belongs to the family Palmae (Arecaceae), is one of mankind's oldest cultivated plants, and is widely cultivated in the Arabian Peninsula [25,26]. Specifically, it is produced largely in hot arid regions, such as Gulf Cooperation Council (GCC) countries [27–29]. The estimated total production of Dates in the GCC countries is  $2.34 \times 10^6$  metric ton per year, with the majority used for human consumption (51%). Animal consumption constitutes 22% of the total production, while 23% of the total production is considered to a waste residue (500,000 metric tons), and represents both an economic and environmental burden. With good waste management plans from major agricultural Date producers, this cheap and abundant waste biomass stream has potential for use in biodiesel production. Compositional analysis of Date pits by Farsi et al. found the following constituents: phenolics; (3102–4430 mg gallic acid equivalents/100 g), protein (2.3–6.4%), fats (5.0–13.2%), dietary fiber (22.5–80.2%), moisture (3.1–7.1%), ash (0.9–1.8% ash) and antioxidants (580–929  $\mu\text{m}$  trolox equivalents/100 g) [30,31]. Furthermore, lipids extracted from Date pits have been reported as having the following fatty acid profile: lauric acid 13–25.8%, palmitic acid 6–13%, oleic acid 11.9–58.8% and myristic acid 7–13% [32].

In this paper, an investigation as to the effect of parameters such as extraction temperature, time and solvent to seed ratio on the extraction of oil from Date pits has been made, utilizing a method of response surface methodology (RSM), the use of which in this context has not previously been reported. Furthermore, biodiesel was produced from the extracted Date pit oil, with measurement of the fuel properties also undertaken.

## 2. Materials and methods

### 2.1. Materials

Date pits (*Phoenix dactylifera* L.), collected from a local "Dates" farm in Muscat, were first cleaned and washed with water in order to remove dirt and pulp. The Date pits (15–20 wt% moisture content) were then sun dried for 6–7 days, followed by overnight drying in an oven at 70 °C to completely remove Date pit moisture content. A mechanical grinder was subsequently used to grind and sieve the pits to obtain a discrete particle size of 250–300 nm. All chemicals and reagents used throughout this work were of analytical grade and were obtained from Sigma Aldrich (Germany) and Merck (Germany).

### 2.2. Oil extraction process and experimental design

Soxhlet extraction was undertaken with the dried Date pit powder, following the AOCS Official Method Am 2-93 [33]. The oil content was extracted from Date pits powder, briefly, a fine ground sample was weighed into a paper thimble (43 mm, Whatman International Ltd., Maidstone, England) and covered with a piece of cotton before placing it in a Soxhlet extractor assembly. The mass of oil extracted by the Soxhlet process was calculated as a percentage by the following formula;

$$\text{Oil (\%)} = \frac{(\text{wt. of predried sample} - \text{wt. of extracted sample})}{\text{wt. of predried sample}} \times 100 \quad (1)$$

To investigate the effect of various process parameters on the extract yield, experiments undertaken were selected using central composite design (CCD) a mode in RSM (Response Surface Method-

ology), using Design-Expert 9.0 (Stat-Ease, Inc) software. The independent variables selected for consideration were temperature, solvent to seed ratio and time, while the yield of extracted oil was chosen as the response variable. Table 1 shows the extraction process conditions and the oil yields obtained; a total of 20 experiments were required in accordance with the CCD methodology, including experiments covering the full range of independent variables, of which 14 were factorial points and 6 were on center points. The range of independent variables was coded into low (−1) and high (+1) levels, with the 6 center points repeated to ensure that any kind of experimental error was avoided. In addition, the experiments were performed in a random order so as to decrease the possibility of errors arising from systematic trends in the variables. The software gave 20 runs to support the data in CCD and following equation was used to calculate the number of experiments (N).

$$N = 2^n + 2n + n_c = 2^3 + (2 \times 3) + 6 = 20 \quad (2)$$

where  $n$  is the number of independent variables and  $n_c$  is the number of repeated runs.

### 2.3. Statistical analysis

The statistical analysis of the experimental data obtained was performed using a response surface methodology (RSM). The interaction between the response variable (oil yield) and independent variables for oil extraction were correlated by a model as described in Eq. (3):

$$y = \beta_0 + \sum_{i=1}^k \beta_{ii}x_i + \sum_{i=1}^k \beta_{i-1}x_i^2 + \sum_{i=1}^k \sum_{j=i+1}^k \beta_{ij}x_{ij} + \varepsilon \quad (3)$$

where  $y$  represents the quantity of oil extracted while  $\beta_0$ ,  $\beta_{ii}$  and  $\beta_{ij}$  are the model coefficients and  $x_i$  and  $x_{ij}$  are coded factors (independent variables).

The significance of the model was evaluated by analysis of variance (ANOVA) in which a  $p$ -value (probability value) of less than 0.05 was considered significant with 95% confidence, and the coefficient of determination,  $R^2$ , and lack of fit was assessed to ensure the predicted quadratic model was the most suitable for the experiments undertaken. A parametric study was carried out based on 3D and contour plots which were obtained using the predicted model, and which show the interaction between two independent

**Table 1**  
Experimental plan for soxhlet extraction of Date pits.

Run	Temperature (°C)	Solvent:seed (wt/wt)	Process time (h)	Oil yield (wt%)
1	50	2:1	5	10.6
2	60	3:1	7	14.7
3	50	3:1	6	12.4
4	70	2:1	5	14.9
5	60	3:1	6	14.4
6	70	4:1	5	16.1
7	60	3:1	6	14.7
8	60	3:1	5	14.4
9	50	2:1	7	11.5
10	70	4:1	7	16.5
11	60	3:1	6	14.1
12	50	4:1	7	13.2
13	70	2:1	7	15.1
14	50	4:1	5	12.5
15	60	3:1	6	14.3
16	60	4:1	6	15.1
17	70	3:1	6	15.9
18	60	3:1	6	14.2
19	60	2:1	6	13.8
20	60	3:1	6	14.6

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