Application of Response Surface Methodology and Central Composite Rotatable Design for Optimization of Supercritical Fluid Extraction of Lipids from Date Pits

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Abstract

Tons of date pits are being produced every year in the United Arab Emirates as by products in the process of making date syrup and other related products. At present, date seeds have very limited use. Date seeds may have extractible high value-added components that are sensitive to high heat as is the case during steam distillation. Application of supercritical carbon dioxide may offer a unique opportunity to extract some of these compounds. Central Composite Rotatable Design (CCRD) and Response Surface methodology are used to optimize parameters for supercritical fluid extraction of lipids from date pits.

The effects of pressure, temperature and CO_2 volume on the extraction yield of date pit oil have been investigated. The temperature tested ranged from 33 to 92 ^oC (33, 40, 60, 80 and 92 ^{o} C) while the pressure tested ranged from 132 to 468 bars (132, 200, 300, 400 and 468 bars). The volumes of CO_2 tested were 66, 100, 150, 200 and 234 ml. Results indicate that at any particular temperature, increasing the pressure will increase the yield. With respect to temperature, the effect depends on the pressure applied. Under low pressures, increasing the temperature tends to decrease the yield. However when the pressure is high the yield tends to increase with increasing temperature. Carbon dioxide volume showed positive effect within the range studied.

Introduction

The date plant (*Phoenix dactylifera* L.) carries several economic and social benefits to the people of the Arabian region. The United Arab Emirates is one of the very important date growing countries. The date population is estimated to be over 40 million trees and around 500,000MT tons of date is produced annually [1]. The fruit of date palm is composed of a fleshy soft part and a very hard seed (pits). The fleshy part is consumed fresh or processed into other products such as date syrup and date juice. In recent years, different kinds of chocolate coated date and date confectionary are becoming very popular in the market. However, apart from being used mainly as animal feed, the seeds have very little economic benefits. Compositional analysis of date seed has shown that depending on the variety, date seed may contain up to 10% of oil [2]. Date seed oil is rich in phenolic compounds, tocopherol and sterols that could be of great use for the cosmetics, pharmaceutical and food industry [3&4]. Date seeds are also known for their health and nutritional benefits (2.3–6.4% protein, 5.0–13.2% fat, 0.9–1.8% ash and 22.5–80.2% dietary fiber) [5].

Extraction of this oil and studying its chemical characteristics may reveal important value added benefits that can be derived from the pits. It is well known that conventional extraction techniques have several disadvantages that people are continuously looking for new and innovative techniques that are less destructive to people and the environment . In recent years, supercritical fluid extraction (SFE) has become an alternative to the conventional extraction techniques. This is mainly because with this technology it is possible to extract heat sensitive compounds and avoid any toxic solvent residues in the product [6&7]. A number of workers have used SFE process to obtain essential oils from different species such as mint, oregano, basil, thyme, parsley, chamomile, sage, and other medicinal plants [8&9]. The objectives of this study are to investigate the suitability of supercritical carbon dioxide extraction technique to extract date seed oil and to optimize the extraction processing condition for maximum yield.

Material and method

Seed material

Commercial dates of 2008 harvest were bought from local market in Al –Ain, United Arab Emirates. The flesh was separated manually, washed and dried at 50 ^{o}C for 12 hours. The sample was milled in a heavy-duty grinder to pass 850 µm screens and then preserved under refrigerated conditions.

Lipid extraction and preservation

Lipid extraction was carried out using ISCO series 2000 SCF Extraction System (SFX 220) consisting of two 10-ml stainless cartridges connected to a 260 ml capacity syringe pump and a controller system (ISCO 260D). Operating pressure of the pump ranged from 1 to 515 bars with an accuracy of 0.5%. The sketch of the diagram is shown in the Figure 1.

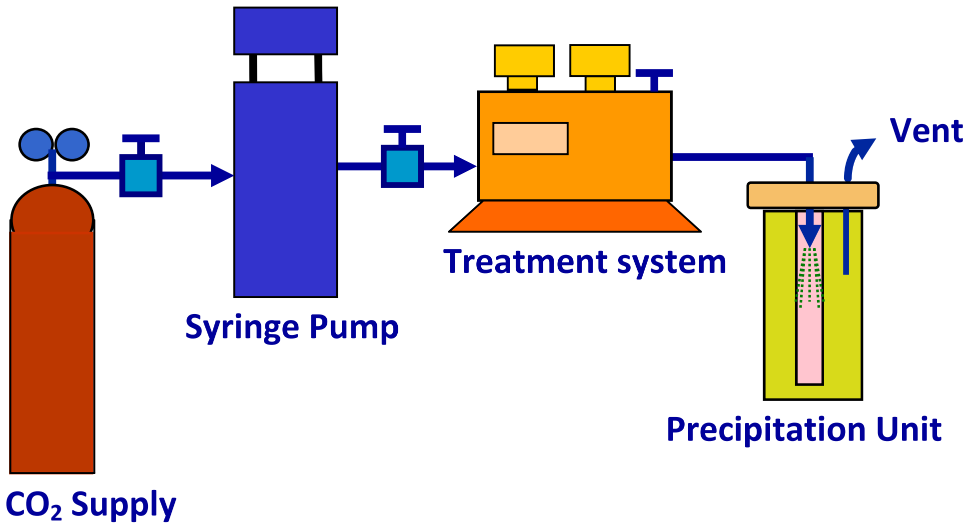

Figure 1: The diagram of the extraction system

Five gram of dry sample was put in the cartridge and a known amount of carbon dioxide was passed at the required pressure and temperature at constant flow rate of 1.5 ml/min. The extract was collected in a small vile submerged in cold methanol at -18 ^{o}C and the extraction efficiency was calculated as reported in [9] using equation no. 1:

$$\text{Extraction efficiency} = \frac{\text{mass of extract}}{\text{Mass of sample}} \times 100 \qquad [1]$$

Experimental design

Central Composite Rotatable Design (CCRD) and Response Surface methodology were used to optimize extraction parameters. The CCRD consisted of three factors, and two levels temperature (40 and 80 ^{o}C), pressure (200 and 400 bar) and volume of carbon dioxide used (100 and 200 ml). The matrix is shown in the table below.

Coded variables			Un coded			
X1	**X2**	**X3**	**Temperature (°C)**	**Pressure (bar)**	**Volume (mL)**	**Yield (%)**
-1	-1	-1	40	200	100	4.96
1	-1	-1	80	200	100	1.67
-1	1	-1	40	400	100	5.42
1	1	-1	80	400	100	6.33
-1	-1	1	40	200	200	5.59
1	-1	1	80	200	200	1.53
-1	1	1	40	400	200	5.60
1	1	1	80	400	200	6.24
1.6818	0	0	92	300	150	6.05
-1.6818	0	0	33	300	150	5.51
0	1.6818	0	60	468	150	5.79
0	-1.6818	0	60	132	150	0.12
0	0	1.6818	60	300	234	5.74
0	0	-1.6818	60	300	66	5.30
0	0	0	60	300	150	5.58
0	0	0	60	300	150	5.51
0	0	0	60	300	150	5.42
0	0	0	60	300	150	5.31
0	0	0	60	300	150	5.43

Table 1 : Matrix of central composite rotatable design

Data Analysis

Analysis of Variance and multiple regression were conducted by using the excel software.

Results and discussion

The effect of temperature and pressure is shown in the Figure 2. From the figure it can be clearly seen that at any temperature, increasing the pressure will give higher amount of extract. This finding is similar to what has been reported [9], who found that increasing extraction pressure has positive effect on the amount of *trans*-lycopene content from tomato skin. The increased extraction yield at high pressure reflects the effect of density on the solubility of the material in carbon dioxide medium. With respect to temperature, it appears that the overall effect depends on the level of pressure applied. At low pressures, raising the temperature will have a negative effect on the yield, however at moderately high pressures; the yield tends to increase with increasing temperature. This finding was also found to be in agreement with what has been reported in literature. In another study of the effect of pressure and temperature on lycopene yield and antioxidant activity, in terms of extraction yield, the best results were obtained at relatively higher temperature and relatively high pressure [11].

The explanation of this behavior is that as the temperature is raised, the density of the supercritical carbon dioxide decreases and the decrease in density will have negative effect on solubility of the material. Therefore, the low yield at higher temperature and low pressure is mainly due to low solubility of the oil in the extraction medium. At high pressure, the effect of density coupled together with the effect of molecular movement due to the increased kinetic energy as the temperature is raised will override the negative effect of temperature on the density and thus a forward shift in the amount of the yield was observed.

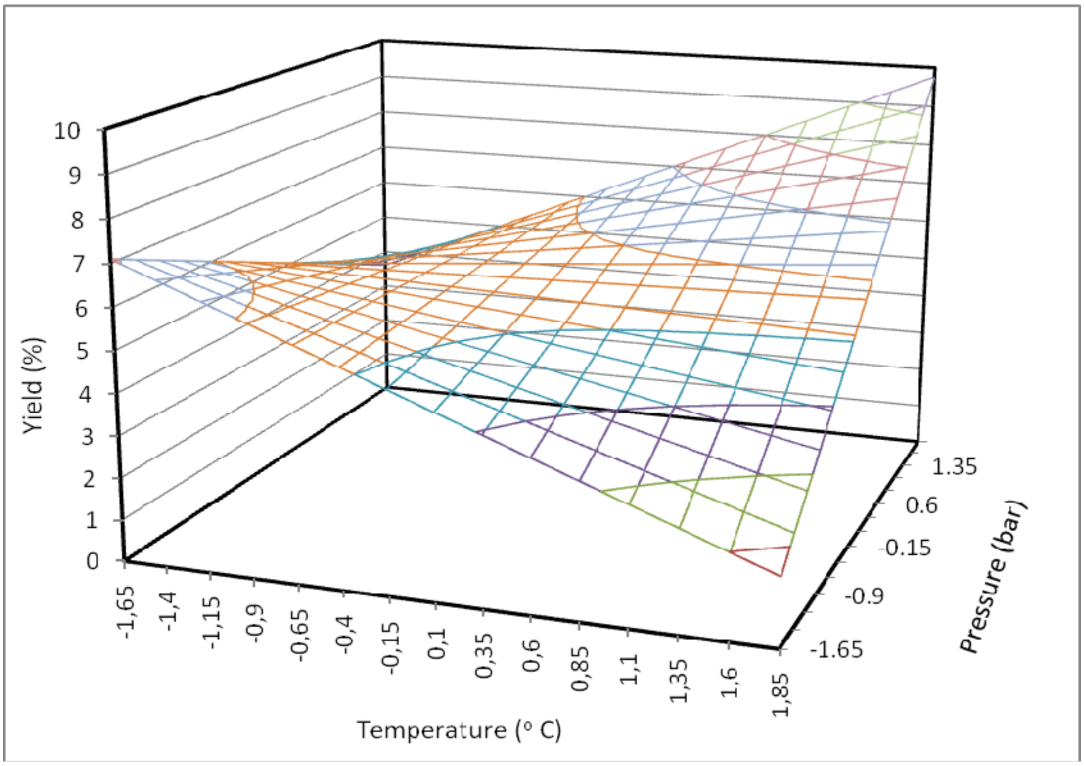

Figure 2: Effect of temperature and pressure on the extraction yield of date pits oil

Figure 3 shows the effect of the volume of CO_2 passed through the sample on the extraction yield. It is well known that at constant pressure, temperate and flow rated, the extraction efficiency will increase with the extension of the extraction time. Therefore logically, the extraction yield will increase with the increase in the extraction volume because in actual fact increasing the volume of CO_2 to be passed means extending the extraction time since the flow rate is kept constant.

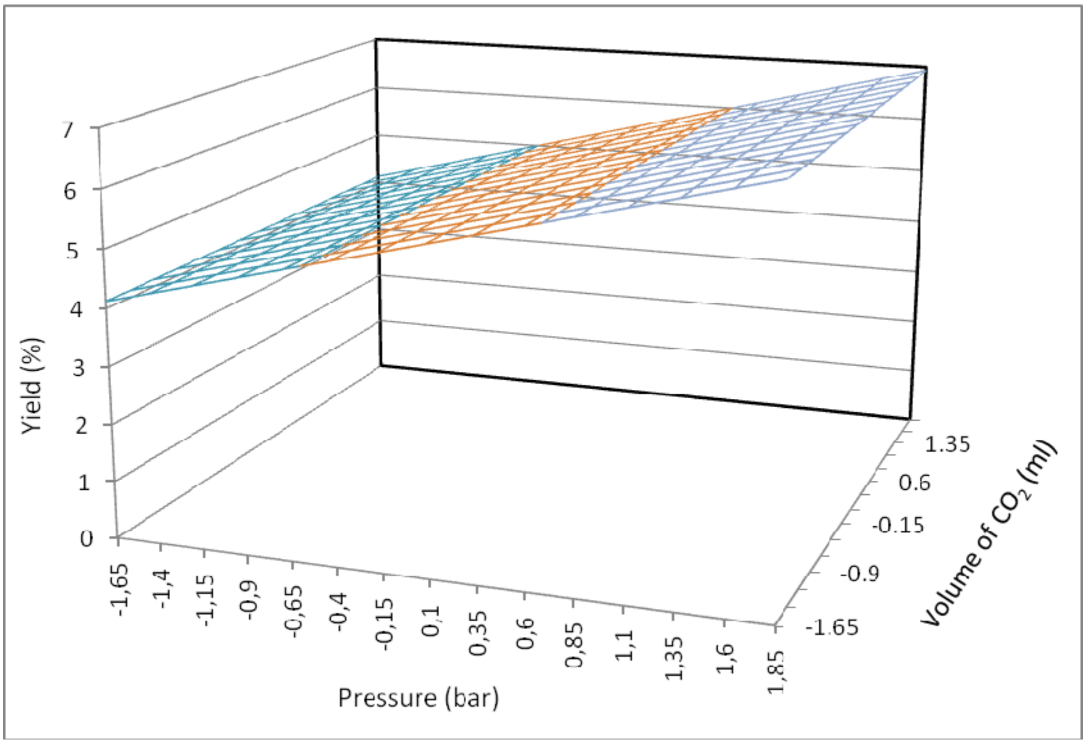

Figure 3: Effect of pressure and volume of CO_2 on the extraction yield

Concussion

The effect of pressure, temperature and volume of carbon dioxide passed on the extraction yield of date pits oil have been investigated using supercritical carbon dioxide extraction technique and central composite rotatable design. Results obtained have shown that pressure and temperature are critical variables that influence the quantity of extracted lipids. The data indicate that at any particular temperature, increasing the pressure will increase the yield. With respect to temperature, the effect depends on the pressure applied. Under low pressures, increasing the temperature tends to decrease the yield due to the effect of density on solubility. However when the pressure is high the yield tends to increase with increasing temperature. Carbon dioxide volume showed positive effect within the range studied.

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