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EFFECT OF PRESSURE, TEMPERATURE AND FLOW RATE ON SUPERCRITICAL CARBON DIOXIDE EXTRACTION OF BOTTLE GOURD SEED OIL

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ABSTRACT

Supercritical carbon dioxide extraction is an important technique to extract medicinal ingredient from the biological source. The bottle gourd seed oil has several uses in pharmaceuticals such as skin therapy in the treatment of benign prostatic hyperplasia and in the cosmetic products such as beauty creams and soaps. The investigation was carried out to extract the functional oil which having very high medicinal value from bottle gourd seed using supercritical carbon dioxide. The results showed that the pressure and temperature had significant effect on the yield of bottle gourd seed oil. However, supercritical CO_2 flow rate had no significant effect on the extraction yield but still high flow rate was essential to ensure optimum mass transfer rate. A 250 µm sized sample gave highest oil yield of about 34.60 per cent at 50 MPa pressure, 333.15 K temperature and flow rate of 15 g/m for 3 hours of extraction.

Key words: Supercritical carbon dioxide, Bottle gourd seed, Functional oil, Pressure, Temperature.

INTRODUCTION

Supercritical carbon dioxide extraction is method of separation of one component from another based on their solubility at different pressure and temperature levels above critical point. The product obtained through this process does not content any residual carbon dioxide at the end of extraction or separation of components and hence the method is treated as green separation method or eco-friendly method. The method is particularly suitable for heat sensitive products such as pharmaceutically active compounds, essential oils and unsaturated fatty acids of lower boiling pints. Supercritical carbon dioxide extraction can be operated under a wide range of operating conditions to selectively extract specific end products or new products with improved functional or nutritional characteristics; to use them as building blocks in creating new formulated foods. Consequently, this technology has become an important separation technique in the field of food and pharmaceutical applications to produce solvent free, thermally liable compounds of interest.

Naturally obtained bioactive compounds are gaining much interest in food and cosmetic industries because of their therapeutic effect over synthetic ones. These compounds are having negligible side effects as compared to synthetically prepared (Sanderson 2011). One of the feed stock, bottle gourd seed which is abundant source of omega-6 fatty acids, also contains some pharmaceutically active compounds used in the treatment of BHP, acne, hyper-seborrhea, alopecia and hirsutism (El-dengawy et al. 2001; Piccirilli *et al.* 2007). The seed oil is also considered anthelmintic and is applied externally for headache.

Bottle gourd seed oil is generally extracted using solvate extraction method (El Dengawy *et.al.* 2001). The conventional solvent extraction has problem of residual solvent in the final product. There are several studies on extraction of high value oil using supercritical carbon dioxide from cottonseed (Bhattacharjee *et.al.* 2007), flax seed (Pradhan *et al.* 2010), neemseed (Tonthubthimthong *et al.* 2001), almond (Passey and Gros-Louis 1993), hazelnut (Ozkal *et al.* 2005), pistachio nut (Palazoglu and Balaban 1998), chia seed (Ixtaina *et.al.* 2010), pumpkin seed (Salgin and Korkmaz 2011) and palm kernel (Zaidul *et. al*. 2007).

Salgin and Korkmaz (2011) reported that operating time required for a certain particle size range increased with decreasing particle size. According to finding for extraction of oil from pumpkin seeds, the extraction yields for 100 min obtained as 0.45, 0.41, 0.24 and 0.16 g oil/g dry seed for the particle size range of 250-600 µm, 600-1180 µm, 1180-1700 µm and 1700-2360 µm, respectively. Salgin and Korkmaz (2011) also found that, the extraction rate were increased initially and found variation in yield from 0.36 to 0.47 g oil/g dry seed with time. Wenli *et al.* (2004), Bernardo-Gil and Lopes (2004) and Mitra *et al*. (2009) have also reported similar effect of temperature on extraction rate with increased time. The extraction yields independent of the supercritical carbon dioxide flow rate with variation in time (Doker *et al.* 2010). But the lack of study on the effect of pressure, temperature and flow rate in connection with different time of extraction of bottle gourd seed oil inspired us and in this context, the investigation was carried out to find out optimum level of parameters to extract bottle gourd seed oil using supercritical carbon dioxide.

MATERIALS AND METHODS

The sample of bottle gourd seeds was collected from local venders of Varanasi Market, Uttar Pradesh. Gaseous carbon dioxide (99% purity) was supplied by Luthra Gas Supplier, Varanasi. Analytical grade n-hexane (Boiling point: 65.5°C), CDH laboratory reagent was purchased from Central Drug House (P) LTD, New Delhi.

The moisture content of bottle gourd seed was minimized by drying at 50°C for 8 hours. The drying operation favors the improvement in the oil extraction rate. After drying, the sample was subjected to grinding operation in a blender to reduce its size upto 250 µm. The ground sample of bottle gourd seeds were kept in the polythene bag and these bags were stored in the dark place to fasten its contact with the light. This helps in reduction in the loss of light sensitive fatty acids (Salgin and Korkmaz, 2011).

The moisture content of dried bottle gourd seeds was determined at 105±5°C (Pradhan *et al.*, 2013). The oil content was determined using Soxhlet apparatus as per procedure given in the AOAC method (1980). SFE 500 model of Thar technologies was used to optimize the process parameters. The model is as shown in the Figure 1.

Figure 1-Semi – continuous supercritical carbon dioxide extraction unit

The ground seed sample to be used in extraction process is placed in the extraction vessel and it is hand sealed. Before keeping the sample in the vessel, it was mixed with the glass beads (2:1 v/v). The gas was then supplied from the cylinder using "*Process Suite software*". The temperature and pressure were also controlled using the computer program. The extraction is carried out until complete extraction took place (maximum up to 4 hours). In each experiment, 100 g sample was charged into the extraction vessel along with glass beads. The extract was collected at every 30 minutes interval i.e. for 30, 60, 90, 120, 150 and 180 min and weighed immediately after collection. The obtained results were divided by the initial weight of the seed sample to convert into percentage of oil content.

The independent variable combinations were selected using orthogonal array design. The L16 (4^3) orthogonal array gave 16 experimental runs for 3 independent variables each with 4 levels. "larger is better" characteristic was selected to get maximum oil yield and S/N ratio was calculated. All statistical analysis was carried out using Minitab 16.1 (Minitab Inc. State college, PA, USA).

RESULTS AND DISCUSSION

The moisture content and oil content of the bottle gourd seed was found to be 10.04 % (wet basis) and 43.40 %, respectively. This value of oil yield is closer to the corresponding value of 39.22 % for kernels of bottle gourd seeds as reported by El Dengawy *et.al,*. (2001). This also suggested that the 250 µm sized particles content mainly kernels while shells were not broken to that size due to that they were higher hardness and removed.

All the selected factors were examined by using 3-factor and 4-level (L16) orthogonal array design. The ANOVA values of S/N ratio showed that pressure and temperature had significant effect on the yield of bottle gourd seed oil while the supercritical carbon dioxide flow rate had no significant effect on it.

Table 1-ANOVA for S/N ratio to analyze parametric effect on extraction yield (%)

Parameters	DF	Seq SS	Adj SS	Adj MS	F
Operating Pressure	3	6.57	6.57	2.187	115.32
Extraction Temperature	3	1.86	1.86	0.62	32.70
CO_2 flow rate	3	0.08	0.08	0.03	1.48
Residual error	6	0.11	0.11	0.02	
Total	15	8.63	-	-	-

The experimental results showed that 50MPa pressure, 333.15 K temperature and CO_2 flow rate of 15 g/min was having highest extraction efficiency. The yield of oil at that combination was highest and was equal to 34.60 per cent. The results also suggested that the method took maximum 3 hour to extract maximum possible oil from the sample. A similar result has also been reported for flax seed (Pradhan *et.al.*, 2010).

EFFECT OF EXTRACTION PRESSURE

The selection of extraction pressure is very crucial as it can affect the extraction yield, rate of extraction and the selectivity of the supercritical fluid also. The orthogonal arrays were designed using four levels of pressure i.e. 20, 30, 40 and 50 MPa. Figure 2 shows the yield of bottle gourd seed oil for different pressure levels at each collection interval.

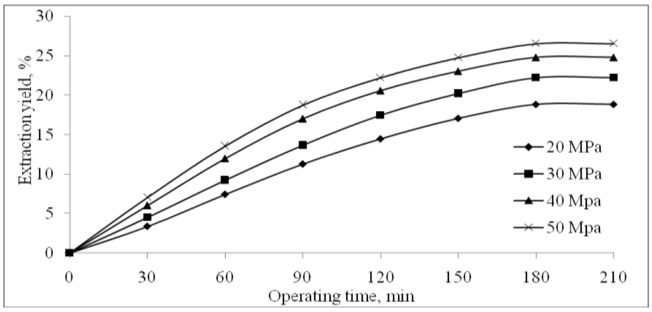

Figure 2 -Effect of extraction pressure and operating time on extraction yield

The increased slopes of the initial parts of the extraction curves with increasing pressure indicated the increase in solubility of the oil in supercritical CO_2. As pressure increased from 20 to 50 MPa, oil yield also increased from 27.93 to 33.83 % (dry basis). However these results showed that the effect of pressure on oil yield was higher as compared to temperature effect. This was due to the fact that the pressure was directly related to supercritical CO_2 density. As the density of solvent increases, the distance between molecules of solvent and solute decreases. Therefore interaction between oil to CO_2 increases which improved the solubility of oil in CO_2. This increase in the solubility of the oil in supercritical CO2 increases the driving force and consequently increasing the mass transfer rate. Hence the extraction yield increases with increase of extraction pressure. The highest oil yield was observed at an elevated pressure of 50 MPa. But further rise in the extraction pressure, increases the cost of extraction process.

EFFECT OF EXTRACTION TEMPERATURE

In this study, the effect of extraction temperature of 303.15, 313.15, 323.15 and 333.15 K on extraction yield of bottle gourd seed oil was studied (Figure 3). In the investigation, effect of temperature on the vapour pressure of solute dominates over effect of temperature on the solvent density. The reduction in the viscosity also caused increase in diffusivity coefficient which enhanced the extraction process. A maximum yield was obtained at 333.15 K. Leo *et al.* (2005) also stated that the enhancement of oil solubility with CO_2 as result of increased temperature causes improvement in the extraction process. A similar trend has been reported by Al-Rawi *et al.* (2013) for nutmeg seed oil.

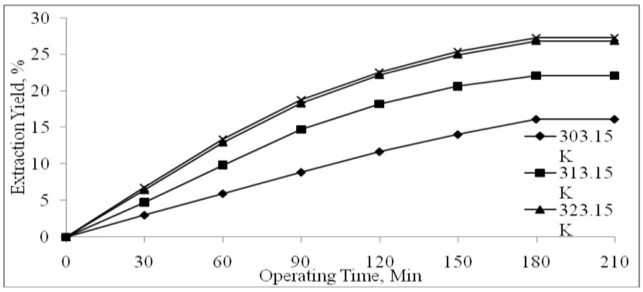

Figure 3-Effect of extraction temperature and operating time on extraction yield

EFFECT OF VOLUMETRIC CO_2 FLOW RATE

To investigate effect of supercritical carbon dioxide flow rate on the extraction of bottle gourd seed oil, ground sample of various sizes were extracted at different pressure and temperature settings as suggested by orthogonal array design with flow rates of 15, 20, 25 and 30 g/min. The oil yield always increased with increasing the flow rate at high pressures or low temperatures. However, when extracted at low pressures or high temperatures, the solubility power of the supercritical CO_2 was not enough to dissolve the oil and a longer residence time would be required for the solvent to become saturated with oil.

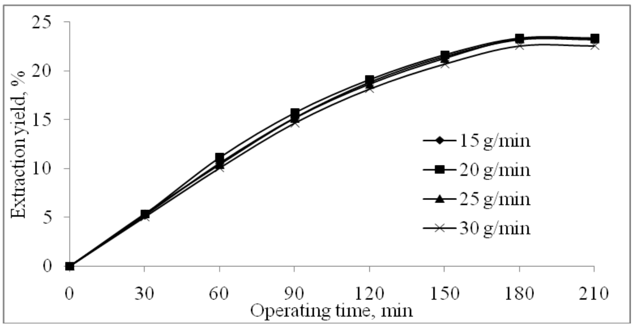

Figure 4- Effect of supercritical carbon dioxide flow rate and operating time on extraction yield

At the beginning of extraction period flow rate was significantly affected the extraction rate (g/h). Hence, higher flow rate would be suggested in order to shorten the extraction time.

CONCLUSION

From the investigation it can be concluded that the supercritical carbon dioxide is an alternative to conventional solvent extraction to extract bottle gourd seed oil. The experimental results showed that selection of pressure and temperature has significant effect on the yield of oil. However, supercritical carbon dioxide flow rate has no significant effect on oil yield. The investigation suggested that 3 hour duration of extraction of 250 µm sized particle at 50 MPa pressure, 333.15 K temperature and 15 g/min flow rate was optimum process parameter combination.

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