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Extraction of Essential Oil from Gomphrena Celosioides by Green Separation Technology

A. K. Tiwari[1], S.R. Geed[2], R.S. Singh[3], B.N. Rai[4]

Department of Chemical Engineering
Indian Institute of Technology (BHU,
Varanasi (UP) India-221005

Abstract: Gomphrena celosioides is an annual herb of family Amaranthaceae.It is source of essential oil particularly alkaloids, tannins, saponins, steroids, glycosides, terpenes and reducing sugars, etc. Semi-continuous supercritical carbon dioxide extraction unit was used to extract the essential oil from the Gomphrena celosioides. Dried plant were subjected to extraction after grinding to particle size of 300μm. The extraction was carried out at four different pressure levels (250,275, 300 and 325 bar), four temperature levels (28, 32, 36 and 40^0C) and four levels of supercritical CO_2 flow rates (10, 15, 20 and 25 g/min). The highest essential oil was obtained at 300 bar, 40^0C and 15 g/min combination of parameters and the highest yield was equal to 4.21gm/100gm. The study showed that thepressure has more significant effect than the temperature while the flow rate was having no significant effect on the yield of Gomphrena celosioides. The study provides an opportunity to pharmaceutical and cosmetic industries to obtain the highly pure essential oil using the optimized conditions.

Keywords: Gomphrena celosioides; supercritical carbon dioxide; essential oil.

1. INTRODUCTION

Supercritical Fluid Extraction (SFE) is one of the methods that can selectively extract specific components. This method has several privileges such as the simplicity of the solvent recovery from the extracted material, the minimization of losses of materials, high purity of the product, as well as the retention of volatile constituents (Ibanez *et al*., 1999). On the other hand, and due to high investment requirements comparing to conventional or traditional methods such as solvent extraction and steam distillation of the products like rosewater, the method is paid less attention. The selective advantage of the extraction method is the flourishing one. Such advantage can easily be achieved depending on the operating conditions. Therefore, this method cannot be feasible when only one compound is extractable in which case the conventional method is more likely to be used. Low operating cost is another advantage of the supercritical extraction. The extraction of pharmacologically active compounds from herbal plants is one of the most critical steps in natural products research. Many parameters affect the extraction procedure. Briefly, the efficiency of the extraction depends on the nature of the sample matrix, the analyte to be extracted and the location of the analyte within the matrix (Luque de Castro *et al*., 2006; Mustafa and Turner, 2011).

In this study Gomphrena celosioides plant is used as the raw material for extraction of essential oil. Gomphrena celosioides is a weed herbs from family Amaranthaceae. Over of 140 species of the same family exist in America, Zimbabwe, Nigeria,India, particularly in western and eastern-ghats, including 46 in Brazil. Very few species are present in East and West Africa (Vieira *et al.*, 1994). This weed of lawns, vacant lots and fields, was probably introduced in West Africa where it is now widespread. In South America, it is used as abortives (Burkill, 1984), in Nigeria it is used for the treatment of dermatological problems (Onocha *et al*., 2005). In Benin, traditional healers use this plant in the treatment of many diseases including liver diseases, malaria and dysmenorrhea (Adjanohoun *et al*., 1989). In India it is used as anti-fungal and anti-biotic in rural areas. Vieira (1994) have demonstrated the analgesic, tonic, carminative and diuretic properties of this plant. Recently, (Dosumu *et al*.,2010) reported its antimicrobial and anti-helminthic properties. The work of (Botha *et al.*, 1986) revealed the presence of saponins, steroids, amino acids, non-reducing sugars, phenols and flavonoids in this plant. However, little information exists on hepatoprotective properties of this plant. It is therefore important works to be undertaken in order to provide a scientific basis for using this plant in the treatment of liver diseases. (Rauen and Schriewer, 1971) have shown that silymarin administered orally opposes the increase in serum transaminases due to poisoning by tetrachloride carbon.

1. MATERIALS AND METHOD

The *Gomphrena celosioides*used in experiment were collected from IIT (BHU) campus Varanasi, Utter Pradesh (India). Carbon dioxide having purity 99.99% is supplied by Luthra Gas Supplier, Varanasi.

1.1. Sample preparation

The Gomphrena celosioides plants were dried at 40 ± 5^{0}C for about 24 h. The dried plants are ground in a blender to uniform particle size 300 µm. The particle size was determined using standard I.S. sieve. The primarily study showed that smaller particle size have higher extraction yield because of large surface area available for mass transfer. Hence the particle size (300 µm) was selected. Samples were kept in the polythene bags which were stored in the refrigerator at 4 ◦C till experiment begins. Because of smaller particle size, large surfaces were exposed to light which enhanced loss of light sensitive essential oils. Hence, the samples were kept away from light before experiment. All the experiments were carried out at room temperature (27^{o}C).

2.2 Experiment set up

Supercritical CO_2 extraction of *Gomphrena celosioides* plant was carried out using semi-batch supercritical extraction equipment (SFE 500 Model, Thar Technologies, Inc., Berlin, Germany as shown in fig.1).The extraction temperature, pressure and CO_2 flow rate was adjusted as per experimental design using Process Suite software. Automatic back pressure regulator (ABPR) was used to regulate the required pressure in extraction vessel. The pressure gauges were placed at extraction and separation vessel to ensure correct pressure settings. Manual pressure regulator was provided at separator end to release CO_2. The fluctuation of pressure and temperature in both vessels (extraction and separation) was under control. It was numerically equal to ±5 units for both. The sample and glass beads (2 mm diameter) were taken in proportion of 2:1 (v/v). Then the mixture was charged in to the extraction vessel. Glass beads were used to reduce packing density and to increase solvent diffusion. The extraction is carried out for 2 h to ensure complete oil extraction for each experiment. In each experiment, 100 g sample was charged into the extraction vessel along with glass beads. The extract was collected at every 30 min interval i.e. for 30, 60, 90 and 120 min and weighed immediately after collection.

Fig.1. Experimental set up

2. VARIOUS STAGES OF GOMPHRENA CELOSIOIDES EXTRACTION:

Plant was collected and dried at a temperature of 40 ^{0}C. The dried plant is shown in fig.2. The plant was grind and the powdered form is obtained which is shown in fig.3. The black colour oil and a light yellow colour waste was obtained in the extraction unit, which is shown in fig.4 and fig.5 respectively.

Fig.2. Gomphrena Celosioide plant (Dried)

fig.3. Gomphrena Celosioides after grinding

Fig.4. Gomphrena Celosioides oil extracted

Fig.5. Gomphrena Celosioides extracted waste

3. RESULTS AND DISCUSSION

The experiment was performed at different temperature pressure and flow rate. The data as shown in table 1

Table1: experimental data.

Sl. no.	Temperat -ure (^0C)	Pressure (bar)	Flow rate (gm/min)	Extraction yield gm/100gm
1	28	250	10	3.4
2	28	275	15	3.35
3	28	300	20	3.39
4	28	325	25	3.7
5	32	250	15	3.84
6	32	275	20	3.39
7	32	300	25	3.85
8	32	325	10	3.97
9	36	250	20	3.9
10	36	275	25	3.85
11	36	300	15	4.1
12	36	325	10	4.15
13	40	250	25	4.05
14	40	275	20	4.12
15	40	300	15	4.21
16	40	325	10	4.19

4.1 EFFECT OF PRESSURE ON OIL EXTRACT

In this process, the effect of extraction pressure on oil extracted is studied for four different pressure (250, 275, 300 and 325 bar) and at 40^oC temperature and 15 gm/min CO_2 flow rate. Fig.6 shows the effect of pressure on the yield. The results of the process shows that there is a significant variation in the oil extracts using different pressure levels. As pressure increased from 250 to 300 bar, oil extract also increased from 4.05gm to 4.21gm. This was due to the fact that the pressure was directly related to SC-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 CO_2 in oil. In this study highest extraction oil is obtained at an elevated pressure of 300 bar. (Ills *et al*.,1999)observed that increase of pressure increases the extraction of oil.

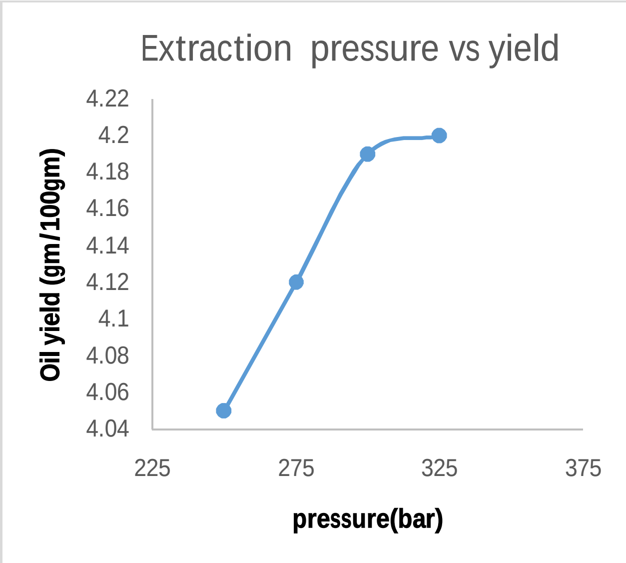

Fig.6. extraction pressure vs yield

4.2.Effect of temperature on oil extract

The effect of extraction temperature on oil collected is determined at four different temperature levels viz. (28, 32, 36 and 40^{0}C) at constant pressure 300 bar and 15gm/min CO_2 flow rate. The effect of temperature on oil collected is more complex to predict than that of pressure because of its two opposite effects. Primarily, increase in temperature may reduce solvent density, thus the solubility of oil decreases. On the other hand, temperature increment raises vapor pressure of solute which may increase solubility of the solvent (Couto *et al.*,2009). Moreover, elevated temperature could increase the mass transfer of oil from the sample. Thus it can be concluded that either solvent density or solvent vapor pressure are important factors in extraction process. The results showed that solvent vapor pressure overcame the effect of solvent density and significant improvement in oil yield with increased temperature (Fig.7). It was observed that viscosity of SC-CO_2 was inversely proportional to extracted temperature. The reduction in the viscosity caused increase in diffusivity coefficient which enhanced the extraction process. Highest average oil collection at 40^{o}C is 4.21gm.has been reported for Gomphrena celosioides plant. In SC-CO_2 extraction, solubility of a solute is depending on the volatility of solute and the solvating effect of solvent. According to (Miller *et al.*,1997), at higher pressure, solvating power increases. Thus it develops a positive linear relationship between solubility and temperature, regardless of the low solvent density. A similar behavior was reported for SC-CO_2 extraction of pumpkin oil (Salgin and Korkmaz ,2011) and flax seed (Pradhan *et al.*,2010).

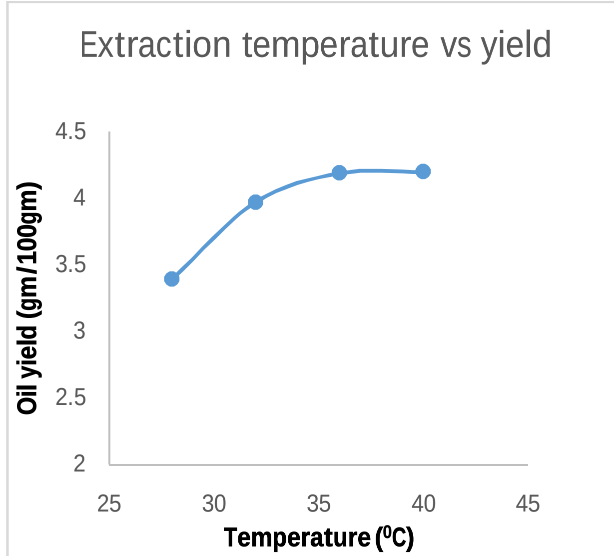

Fig.7. extraction temperature vs yield

4.3. Effect of CO_2 flow rate

The effect of volumetric SC-CO_2 flow rate on extraction oil collected is investigated at four different levels (10,15, 20 and 25 gm/min) at temperature 40^{0}C and pressure 300 bar. At high pressure and low temperature, the oil yield always increased with flow rate. However at low pressures and/or high temperatures, initially oil collected increased and then decreased with increase in flow rate (Zhao and Zhang.,2013). Solubility of solute is important parameter which controls the extraction percentage. Mass transfer increased with increased in solvent flow rate until it attained an asymptotic value.

However, the residence time of solvent reduced with increasing volumetric flow rate. At the beginning of extraction period flow rate was significantly affected the extraction rate (gm/min). Hence, higher flow rate would be suggested in order to shorten the extraction time. Although the solvent flow rate alone had no significant effect, it had significant effect in combination with the extraction pressure and temperature. Fig.8 shows that there was no significant effect of solvent flow rate on oil collected. A similar behavior was reported by(Jokic *et al.*,2012)for SC-CO_2 extraction of soybean oil.

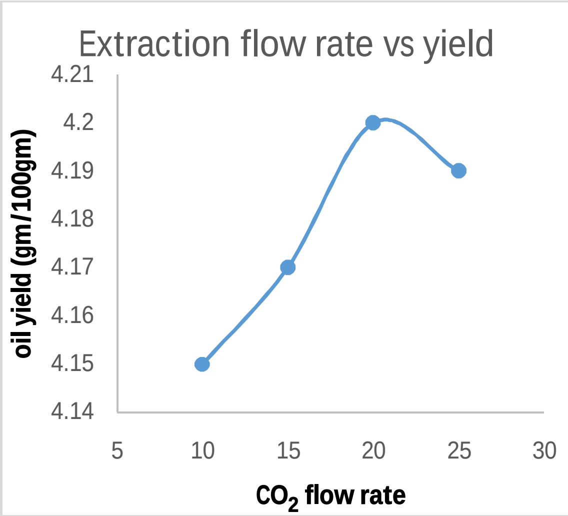

Fig.8. extraction flow rate vs yield

4. CONCLUSION

Day by day increasing the interest of bio-active ingredients in the field of pharmaceutical and cosmetics, increases the demand of pure product. The super critical fluid extraction process gives almost pure product as there is no need of additional solvent to extract the bio-active ingredients from the herbs. The highest yield of essential oil was obtained at 300 bar, 40 and 15 g/min combination of parameters and the highest yield was equal to 4.21gm/100gm. The study showed that the pressure has more significant effect than the temperature while the flow rate was having no significant effect on the yield of Gomphrena celosioides. The study provides an opportunity to pharmaceutical and cosmetic industries to obtain the highly pure essential oil using the optimized condition. The presence of alkaloids, tannins, saponins, steroids, glycosides, terpenes and reducing sugars provides a good anti-fungal and anti-bacterial activity.

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