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Comparative Evaluation of Extraction Methods for Extraction of Essentioal Oil from *Foeniculum Vulgare*

Jigar B. patel[a], Bhavna Patel[b], Rajendra K. Patel[a], Bhavesh H. Patel[a]

[a] K. B. Institute of Pharmaceutical Education and Research, Kadi Sarvavishwavidyalaya, Gandhinagar, Gujarat, India.
[b] P. G. Department of Pharamceutical Science, Sardar Patel University, Vallabh-Vidyanagar, Gujarat, India.

ABSTRACT:

Foeniculum vulgare, commonly known as fennel, is used as carminative and purgative. The extraction of fennel powder was carried out by traditional method like Hydrodistillation (HD) and newer methods like Supercritical Fluid Extraction (SCFE) and Improved Microwave Assisted Extraction (IMAE) technique. Oils extracted by different methods were determined by GC-MS method. The total content fraction of determined compounds were100%, 85.3 % and 105.1 % for supercritical fluid extraction, Improved microwave assisted extraction and hydrodistillation respectively. The extraction times were 150 minutes, 25 minutes and 210 minutes for HD, SCFE and IMAE respectively. The yield was 0.88, 0.5 and 0.4 % for HD, SCFE and IMAE respectively.

Keywords: *Foeniculum vulgare*, Hydrodistillation, Supercritical fluid extraction, improved microwave assisted extraction.

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For Correspondence:

Mr. Jigar B. Patel

K. B. Institute of Pharmaceutical Education and Research,

Kadi Sarvavishwavidyalaya,

Gandhinagar, Gujarat, India.

Email: jigas2181983@gmail.com

INTRODUCTION:

Foeniculum vulgare, commonly known as Fennel, is a plant species belonging to Umbelliferae family. Generally it is used for its carminative and purgative effect. It is a constitutent of grip water. Essential oils are generally present at low concentration and their composition is complex. There are so many conventional methods to extract it before analysis e.g. hydro-distillation (HD), steam distillation, Soxhlet extraction, and solvent extraction. These compounds are suseptical to chemical changes and thermally sensitive.[1–4] There are certain disadvantages of these methods like losses of some volatile compounds, low extraction efficiency, degradation of unsaturated or ester compounds through thermal or hydrolytic effects, toxic solvent residue in the extract etc. [5-6] These kind of disadvantages have led to the consideration of the use of new eco-friendly technique in essential oil extraction which use less solvent and energy, such as supercritical fluids, ultrasound and microwave.

MATERIALS AND METHODS

Seeds of *Foeniculum vulgare* were purchased from the local market of Mehsana of North Gujarat. It was crushed and passed through the sieve with aperture size of 0.4 mm. Carbonyl iron powder (99.16 % Iron content, 3.5 µm) was procured as gift sample from Puneet Laboratories, Mumbai.

Instrumentation and condition

Hydro-Distillation

Powdered seeds (500 g) were extracted with Clevenger-type apparatus according to the European Pharmacopoeia [15] with 6 L of water for 4.5 h (until no more essential oil was obtained). The essential oil was collected, dried under anhydrous sodium sulphate and stored at 0 ◦C until used.

Table 1: Results and comparison

Sr No	RT	Compound	SCFE	IMAE	HD
			% Content		
1	5.75	α-Limonene	2.33	3.1	5.4
2	7.18	L-fenchone	5.81	6	15
3	9.417	Methyl chevicol [(1-methoxy-4-(2-propenyl) benzene)]	2.87	1.2	10
4	10.48	Anisic aldehyde (4-methoxy benzaldehyde)	1.64	1	0.5
5	11.1	Anethole [(1-methoxy-4-(1-propenyl)-benzene)]	83.82	70	70.2
6	12.02	palimitic acids	0.04	n	n
7	12.4	γ-terpenene	0.71	1.1	1.3
8	20.76	Myrcene	0.98	1.3	0.9
9	28.55	Caprinone (10-nonadecanone)	1	0.9	0.3
10	28.97	Unidetified mass	0.8	0.7	1.5
Total content fraction of determined compounds			100	85.3	105.1
Extraction time			2.5 h	25 min	3.5 h
% yield			0.88	0.5	0.4

Superctritical Fluid Extraction

25 kg of the pulverised Fennel powder was feeded in the extraction chamber of supercritical fluid extractor (Flevex Aromats India Ltd, Pune, India). It was mixed with the pressurized CO_2 supplied from the CO_2 gas cylinder. The pressure was maintained constant (190 kg) by the pressure valve. The temperature was maintained 42^0 C. The material was allowed to agitate for 2.5 hours. Then the pressurized CO_2 was transferred to second chamber where the pressure was reduced to liberate CO_2 which was recycled. The extract (220 g) was stored at 0^OC until analysed further.

Improved Microwave Assisted Extraction

The extraction was carried out by using the modified laboratory microwave oven (Model-180F, Catalyst, Pune). 500 g of powdered material was moistened with water. It was heated using a fixed power of 140 W for 30 min with 20 g of CIP without added any solvent or water. A cooling system outside the microwave cavity condensed the distillate continuously. Condensed water was refluxed to the extraction vessel in order to provide uniform conditions of temperature and humidity for extraction. The extraction was continued at 100 ◦C until no more essential oil was obtained. The essential oil was collected, dried under anhydrous sodium sulphate and stored at 0 ◦C until used.

GC-MS analysis

It was carried out using Elite-5 model of Perkin Elmer, USA using fused-silica-capillary column with a stationary phase P-5MS (30m × 0.32mm × 0.25mm film thickness). The carrier gas was He with the flow rate of 0.7 mL/min and injection volume was 0.4µL. The injection temperature was 250 ◦C. Oven temperature progressed from 80 to 280 ◦C at 10◦C/min, holding at 280 ◦C for 40 min. The ionization mode used was electronic impact at 70 eV. The data was analysed by literature survey and NIST library.

RESULTS AND DISCUSSION

The results were compared considering the total content fraction, content fraction of major constituents, extraction time, % yield as evaluation parameters in table 1. The total content fraction of determined compounds were 100%, 85.3 % and 105.1 % for supercritical fluid extraction, Improved microwave assisted extraction and hydrodistillation respectively. Anethole is considered as major constituent. The % content of it was found to be 83.82%, 70 % and 70.2% by

the SCFE, IMAE and HD method respectively. The minor constituents were also present in extract of SCFE but not found in extract of IMAE and HD. The extraction times were 150 minutes, 25 minutes and 210 minutes for HD, SCFE and IMAE respectively. The yield was 0.88, 0.5 and 0.4 % for HD, SCFE and IMAE respectively. The comparison reveals that HD method is time consuming, costly and produces poor quality oil. SCFE produces double yield of oils compared to IMAE and HD methods. The oxygenated product is also less in comparison to these two methods. The SCFE is the best suitable method for industrial application due to high yield, good quality of oil, cost and ecofriendliness.

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