MICROWAVE EXTRACTION OF FRANKINSCENCE ESSENTIAL OIL

The conventional and most popular method to extract the essential oil of Boswellia species is the traditional hydrodistillation (HD). Although it requires a low initial investment, this technique entail many limitations that can be overcome by a novel, fast and efficient extraction technique, the solvent-free microwave extraction (SFME) performed by the Milestone ETHOS X family. SFME was applied on Boswellia serrata oleo gum resins and compared to HD. ETHOS X demonstrated to be the fastest and the most efficient extraction technique, maximizing the quality of the organoleptic profile of the final oil. The outcome of SFME is a product of higher antioxidant activity achivied through a high profitability process.

INTRODUCTION

Gum resins from Boswellia species, also known as frankincense, have been used as a major ingredient in Ayurvedic and Chinese medicine to treat a variety of healthrelated conditions. Both frankincense chemical extracts and essential oil prepared from Boswellia species gum resins exhibit anti-neoplastic activity, and have been investigated as potential anti-cancer agents. The anti-cancer activity is mediated through multiple signaling pathways. In addition, frankincense essential oil overcomes multicellular resistant and invasive phenotypes of human breast cancer cells. Fast and green Extraction of Frankincense essential oil turns out therefore to be extremely important. This essential oil is obtained from a resin from the bark of a shrub originally from the area surrounding the Red Sea, in Somalia and Arabia. To collect the resin, fine incisions are made in the bark, and drops of sap appear and dry in large, odorous yellow droplets (Xiao Ni, 2012). More than 300 volatiles in frankincense have been reported in the literature. A broad diversity has been found in the qualitative and quantitative composition of the volatiles with respect to different varieties of Boswellia (Michaela Mertens, 2009) (Johannes Niebler, 2016). Also, the abundance of essential oils is highly dependent on distillation process parameters (time and temperature) typical duration of HD may vary between 6 h and up to 12 h (Bikram Singh R. K., 2006). Beside being an energy consumption technique, key labile constituents could be lost during this process at elevated temperatures for

prolonged periods, whereas other compounds might be formed as artifacts. This application report proves that the Milestone ETHOS X is a new effective and green extraction technique which is able to tackle those issues, ending up with premium quality products.

I EXPERIMENTAL

INSTRUMENT

- ETHOS X Microwave Green Extraction of Natural Products
- Stainless-steel fragrances kit set up
- Fragrances extraction vessel 2 L
- Chiller 1 kW

ETHOS X 2.0 and ETHOS XL defer from the ETHOS X only for their production capacity. These two systems are based on the same technology and designed for enhanced production capacity.

SAMPLE AND REAGENTS

- Boswellia serrata oleo gum resin in the form of amber to orange-brown lumps (Mohammad Turk, 2018)
- Distilled water

PROCEDURE

Oleo gum resin lumps bags were assembled in one lot. The size of the lumps was reduced using a grinder at 3000 rpm and a 4 mm sieve. The obtained powder was sieved using a sieve shaker set to a 2 min cycle. Only the fractions between 1 and 2 mm (94% of the sieved powder) were used for the extraction step.

SOLVENT-FREE MICROWAVE EXTRACTION (SFME)

40g of oleo gum resin powder was weighted and a prior rehydration step with a ratio 1:5 material to water was necessary to let the material retrieve the natural amount of water lost over the drying steps before commercialization.

After this step the rehydrated material was placed inside the 2 L Fragrances extraction vessel. Once closed, the reactor was placed inside the ETHOS X cavity and the stainless steel distillation module was assembled. The sample was heated at the power density 1 W/g of solid. After 6.7 min, the aromatic essential oil fraction started to be collected in the graduated burette, above the water layer. The extraction was completed after 47 min when the terpenes were completely extracted. At the end of the extraction, the essential oil fraction was collected from the glass distillation module, frozen for 30 min to remove extra water and then stored in a refrigerator at 4°C before analysis.

CONVENTIONAL HYDRODISTILLATION (HD)

HD was carried out using the same Clavenger-apparatus but using a conventional heater as heating source. The extraction lasted 180 min. At the end of the extraction, the essential oil fraction was collected from the glass distillation module, frozen for 30 min to remove extra water and then stored in a refrigerator at 4°C before analysis.

The essential oil fraction and relative quantification was performed by gas chromatography coupled to mass spectrometry (GC-MS) and data acquisitions Mass spectra peak assignment was based on a comparison with NIST database (NIST MS Search 2.0).

The radical scavenging activity of extract was evaluated by a modified version of the method using DPPH radicals, proposed by Brand-Williams et al. (W.Brand-Williams, 1995) (Mohammad Turk, 2018).

The overall profitability of the two extraction processes HD and SFME have been also compared, taking into account two main direct costs specifically the cost of electrical energy and the purchase price of the raw material using the equation (Mohammad Turk, 2018).

| RESULTS AND DISCUSSION

Table 1: SFME, HD results

	EXTRACTION TECHNIQUE	
PARAMETERS	SFME	HD
Time	47 min	180 min
Extraction Yield	2.4 ± 0.1%	2.4 ± 0.1%
DPPH radical scavenging activity	+51.5%	+25%
Profitability	+4	+2.28

As shown in table 1, SFME and HD show the same essential oil recovery of 2.4 ± 0.1% in yield (w/w). Total extraction time are 47 minutes for SFME and 180 minutes for HD.

GC-MS chromatogram analysis revealed 53 compounds among which 38 were fully identified (Table 2). The mean contents of these compounds in the EO samples were 85.5% for SFME and 87.7% for HD (Table 2). The chemical analysis of Frankincense EO showed that most of the identified molecules belong to monoterpene, oxygenated monoterpene, and sesquiterpene families. The main components were characterized as a-thujene, estragole, α-pinene, cembrene and in less proportions, o-cymene, 3-carene, β-bourbonene, and methyl eugenol. The deviation of proportion from the reference treatment (HD) of olibanum oil compounds obtained by SFME that Mohammad Turk et al. shows, suggests that the microwave treatment enables to obtain essential oils of different chemical compositions from reference extraction methods. Furthermore, the composition depends on the treatment parameters.

The antioxidant activity is defined by the volume of essential oils in microliters required to inhibit 50% of the DPPH radical and compared with lavender essential oil with known antioxidant activity. The antioxidant potency of essential oil extracted by HD showed to be 25% higher while the one extracted through SFME was 51.5% higher than the standard known reference of lavender (Mohammad Turk, 2018).

Mohammad Turk et al. studied also the efficiency of the extraction process by a ratio of income over costs: $Profitability = EO_t P_{EO} / EP_E + m_{RM} P_{RM.}$ The income is determined by the product of the selling price (EO_{price}) and the recovered quantity (EO_t) during an extraction cycle of the Essential oil (EO). The quantities of EO (EOt) and electric energy (E) are time dependent. In this study, the following costs were considered for EO (P_{EO}= 1.63 €/g EO), electric energy (P_E = 0.145 €/kW h), and oleo gum resin (P_{RM} = 5 €/kg) (Mohammad Turk, 2018). According to the equation described in the article, both processes are profitable since the balance between income and cost corresponds to a profitability higher than 1, but SFME is clearly more profitable than HD (4 against 2).

Table 2: GC-MS results for SFME and HD on Frankincense essential oil

N°	COMPOUNDS	Frankincense	
		SFME	HD
1	α-Thujene	23.9	17.2
2	α-Pinene	13.5	11.5
3	Thuja-2,4(10)-diene	0.4	0.2
4	β-Pinene	0.6	0.6
5	α - Phellandrene	0.2	0.1
6	(E)-β-Ocimene	0.2	0.2
7	Terpinolene	0.2	0.1
8	o-Cymene	4.4	4
9	3-Carene	2.3	2.1
10	γ-Terpinene	0.2	0.1
11	α-Thujone	0.7	0.5
12	Sabinyl acetate	0.2	0.2
13	(-)-Myrtenol	0.3	0.3
14	*cis*-Verbenol	0.4	0.4
15	*cis*-Sabinol	**0.6**	**0.5**
16	Thujen-2-one	0.1	0.1
17	*trans*-Sabinene hydrate	0.4	0.4
18	2-Carene	0.2	0.2
19	Estragole	17.3	11.6

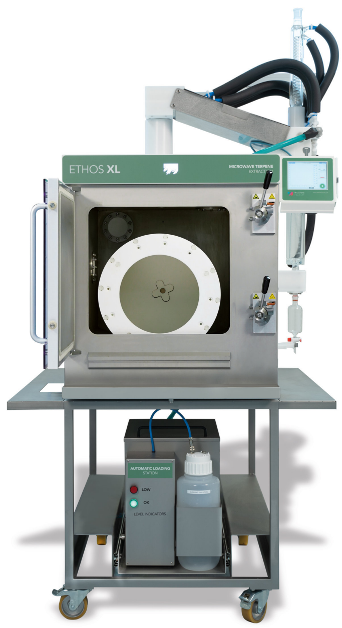
20	(-)-Verbenone	0.2	0.2
21	*p*-Cumic aldehyde	0.1	0.1
22	Bisabolene<(Z) -Alpha->	0.02	0.03
23	Bornyl acetate	0.1	0.1
24	Cyclosativene	0.1	0.1
25	α-Copaene	0.6	0.7
26	β-Bourbonene	1.4	1.6
27	Methyl eugenol	2	2.9
28	β-Ylangene	0.2	0.2
29	β-Copaene	0.2	0.2
30	γ-Muurolene	0.6	0.9
31	α-Muurolene	0.1	0.1
32	δ-Guaiene	2.7	3.3
33	Guaia-1(10)11-diene	0.2	0.2
34	Elemicin	0.8	1
35	Cadinene	0.3	0.4
36	α-Gurjunene	0.3	0.3
37	Verticiol	2.3	1.2
38	Cembrene	5.8	6.2

CONCLUSIONS

The data shown in this work demonstrates that the ETHOS X system is able to maximize the extraction of essential oils from Frankiscence within 47 minutes total extraction time, showing to be the most efficient and rapid process on the market, compared to conventional extraction techniques. The fast processing time permits to obtain an oil which preserves its original chemical composition, sharing tremendous antioxidant properties. Moreover the SFME process showed to be much more profitable than HD, enabling the production of a superior organoleptic profile with muss less energy consumption than the conventional extraction techniques. Milestone's ETHOS X has proven to be a unique and beneficial solution for Frankiscence essential oil producers, allowing them to obtain scents with unmatched quality in very short runs.

Based on the same technology and approach Milestone has developed the ETHOS X 2.0 and the ETHOS XL dedicated to more intense processes. The ETHOS X 2.0 is available with a 15 L reactor and a simplified handling, to ensure fast and easy production capabilities.

The ETHOS XL has been built for medium-large size producers and has a rotating drum with a total capacity of 45 L and semiautomated workflow for more intense production needs.



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