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Sage oil extraction and optimization by response surface methodology

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ABSTRACT

Sage oil extraction is important as most of the components in sage play an important role in the treatment of various diseases. The aim of the present study was to optimize supercritical extraction parameters for sage (*Salvia officinalis* L.) oil yield by response surface methodology. A 3-factor Box-Behnken design was used to generate factor combinations. The optimal conditions for the sage oil extraction yield were 280 °C extraction temperature, 110 min extraction time and 11% (wt/v) plant concentration in ethanol. The validation experiment showed that the actual and predicted values were 49.21 and 47.04 wt%, respectively. The composition of sage essential oil consisted mainly of acid esters together with phenols, limonene, indoles, and pyrroles. These compounds can be used in various industrial applications.

1. Introduction

Sage, a well known aromatic herb, is mainly used as an herbal tea. Besides, this plant may alleviate coughs and bronchial infections (Arranz et al., 2014). It has also high potential for the development of medicinal products as most of the components in sage play an important role in the treatment of various diseases. Previous studies have demonstrated that the sage extracts have been found to possess antioxidant, antimicrobial, anti-inflammatory and antidiabetic activities (Arranz et al., 2014; Bozin et al., 2007; Ehrnhöfer-Ressler et al., 2013; Eidi and Eidi, 2009; Miura et al., 2002). Thus, it is important to extract active components from sage with the use of an appropriate extraction method. Several extraction methods have been applied for the extraction of the active components from sage. In these processes, the research was mainly focused on the optimization of extraction parameters to obtain either the extract yield or the target compound (Ollanketo et al., 2002; Dragović-Uzelac et al., 2012; Roby et al., 2013, Langa et al., 2009).

Ollanketo et al. (2002) reported the extraction of antioxidative components from sage by pressurized hot water extraction, ultrasonication-assisted methanol extraction, hydrodistillation, and maceration with 70% ethanol. Pressurized hot water extraction was the most efficient method for extraction of sage oil. Microwave extraction of polyphenols from sage was carried out by Dragović-Uzelac et al. (2012). The extraction parameters tested in the study were extraction time (3, 5, 7, 9 and 11 min), microwave power (500, 600 and 700 W), and extraction solvent (ethanol:water 30% w/w, acetone:water 30% w/w). The optimal conditions for high content of polyphenols were microwave power of 500 W and extraction time of 9 min. The case of ethanol was similar to acetone. Both solvents showed higher extraction efficiencies than water. Roby et al. (2013) conducted a study concerned with the extraction of phenolic compounds in thyme (*Thymus vulgaris* L.), sage (*Salvia officinalis* L.), and marjoram (*Origanum majorana* L.) using different solvents (methanol, ethanol, diethyl ether and hexane). The yield of extraction and total phenolic content changed depending on the type of solvent used. The highest sage oil yield (23.5%) and total phenolic content (5.95 mg/g) were obtained using methanol as solvent. In another study, supercritical carbon dioxide (Sc-CO_2) extraction of sage oil was studied (Langa et al., 2009). The extraction parameters were temperature (40, 50 °C), pressure (90, 100 bar) and plant particle size (0.3, 0.5, 0.8 mm). The highest sage oil yield was achieved at 40 °C and 90 bar with a particle size of 0.8 mm.

Supercritical fluid extraction (SFE) is an efficient analytical technique to obtain active components and/or essential oils from plants. In SFE, the optimization of extraction parameters (such as plant concentration in solvent, extraction temperature and extraction time) is important to obtain extracts efficiently. Regarding the SFE process, classical optimization is applied to extraction parameters in most previous reports. Recently, the application of experimental design using response surface methodology (RSM) has been widely

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Table 1
Range and levels of independent factors.

Factors	Symbol	Range and levels of independent factors		
		−1	0	+1
Temperature (°C)	x_1	280	300	320
Time (min)	x_2	60	90	120
Plant concentration in ethanol (wt/v, %)	x_3	8	10	12

Table 2
The design matrix and responses for the experimental values.

Run	Variables						Responses
	x_1 Temperature(°C)		x_2 Time (min)		x_3 Plant concentration in ethanol (wt/v, %)		Y_1 Sage oil yield (wt%)
1	280	−1	60	−1	10	0	44.80
2	320	+1	60	−1	10	0	58.80
3	280	−1	120	+1	10	0	50.20
4	320	+1	120	+1	10	0	63.10
5	280	−1	90	0	8	−1	49.80
6	320	+1	90	0	8	−1	71.00
7	280	−1	90	0	12	+1	44.90
8	320	+1	90	0	12	+1	72.00
9	300	0	60	−1	8	−1	51.00
10	300	0	120	+1	8	−1	54.10
11	300	0	60	−1	12	+1	51.50
12	300	0	120	+1	12	+1	54.80
13	300	0	90	0	10	0	51.00
14	300	0	90	0	10	0	52.80
15	300	0	90	0	10	0	49.60
16	300	0	90	0	10	0	50.40
17	300	0	90	0	10	0	51.60

Table 3
Analysis of variance (ANOVA) for quadratic model.

Sources	SS	Df	MS	*F*-value	$F_{0.05}$
Sage oil yield					
Regression	915.58	9	101.73	10.41	3.68
x_1	708.76	1	708.76		
x_2	32.4	1	32.4		
x_3	0.98	1	0.98		
x_1^2	98.94	1	98.94		
x_2^2	12.2	1	12.2		
x_3^2	50.77	1	50.77		
x_1x_2	0.3	1	0.3		
x_1x_3	9	1	9		
x_2x_3	0.01	1	0.01		
Residual	68.41	7	9.77		
Lack of fit	62.52	3	20.84		
Pure error	5.89	4	1.47		
Total	983.99	16			
R-squared 0.93 AdjR-squared 0.84					

SS: sum of squares, Df: degrees of freedom, MS: mean square.

used for optimization of experimental parameters as it is not only time and reagent saving, but it also provides information about the interactions of parameters (Duarte and Duarte, 2011; Leardi, 2009). Experimental design for process optimization via response surface methodology has been studied by many researchers (Bingol and Kulcu, 2011; Liu et al., 2012; Esfahani et al. (2014); Djafarzadeh et al., 2013; Akalin and Karagoz, 2014). Available experimental designs for process optimization include the following: full factorial design (FFD), Plackett–Burman design (PBD), Taguchi design (TD), cental composite design (CCD) and Box-Behnken design (BBD) (Sharif et al., 2014). Among these options, the Box-Behnken design has been widely applied in the optimization of SFE parameters (Turner et al., 2004; Cvjetko et al., 2012; Khajeh, 2011).

Since there is no other study concerning the optimization of SFE extraction parameters for sage oils using multivariate statistics, the present paper provides the optimization of supercritical ethanol extraction parameters. Supercritical fluid extraction of sage was carried out using RSM with the purpose of studying the effects of supercritical ethanol extraction parameters and their interactions on the extract yields. The composition of the selected oil was identified by gas chromatography-mass spectrometry (GC–MC). The identification of the elemental contents of oils was also carried out using an elemental analyzer. The surfaces of sage and residues after the extraction were characterized by a scanning electron microscope (SEM).

2. Materials and methods

2.1. Materials

The sage samples used in this study were harvested in the Aegean region of Turkey. The dried samples were ground and used in the experiments. The ethanol used in the optimization experiments was analytical grade and purchased from Sigma–Aldrich.