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Research Article

Valorization of lavender waste – obtaining and characteristics of polyphenol rich extracts

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Abstract

Bulgaria became the leading producer of lavender oil in the last years. Due to the lower quantity of essential oil large amounts of waste were generated which distilleries usually discard, although the residues are rich source of biologically active substances. The objective of this study was to obtain polyphenol-rich extracts from lavender waste and to investigate their chemical composition and antioxidant capacity. Two wastes (Mirkovo, Bulgaria, 2016) were investigated – steam distilled (SD-L) and CO_2-extracted lavender (CO2-L). The major aroma constituents found were linalool and linalyl acetate – 30.68% and 25.82%, respectively, and the highest concentrations were found in the SD-L. The total flavonoids in CO2-L and SD-L were 2.91±0.11 and 3.72±0.20 mg.g^{-1} dry matter residue, respectively. The higher amount of phenolic acids was observed in the SD-L waste – 2.62±0.19 mg.g^{-1}, compared to 1.39±0.14 mg.g^{-1} dry matter residue for CO_2 extracted lavender. The antioxidant activity of the extracts was investigated by DPPH (SD-L – 355.48±23.12 µmol TE.g^{-1} DW waste; CO2-L – 283.21±17.04 µmol TE.g^{-1} DW waste) and FRAP (SD-L – 427.36±26.54 µmol TE.g^{-1} DW waste; CO2-L – 311.29±18.17 µmol TE.g^{-1} DW waste). Both methods suggested that SD-L residue had higher antioxidant capacity and polyphenol content.
The lavender wastes (SD-L and CO2-L) showed strong antioxidant capacity with potential beneficial effect on addition in foodstuffs. For the first time lavender residues from CO_2-extraction was investigated for its antioxidant activity, polyphenol composition and aroma metabolites, and comparison with SD-L was performed. In general, the results suggested that the lavender waste were promising source of antioxidants.
Keywords: lavender, waste valorization, polyphenols, antioxidant activity, CO_2 extraction

Abbreviations:
ANOVA – analysis of variance; HORAC – Hydroxyl Radical Averting Capacity; BSTFA – N, O-Bis-(trimethylsilyl)-trifluoroacetamide; DPPH – 2, 2-diphenyl-1-picrylhydrazyl; FRAP – ferric reducing ability of plasma; HPLC – high performance liquid chromatography; NIST – National Institute of Standards and Technology; ORAC – oxygen radical absorbance capacity; RI – relative index (Kovats retention index)

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Introduction

The lavender is among the most processed crops by the essential oil manufacturers. The main industrially exploited species are the true lavender (*Lavandula angustifolia* Mill.), lavandin (*Lavandula x intermedia* Emeric ex Loiseleur) and spike lavender (*Lavandula spica* D.C.). Bulgaria, France, UK, China, Ukraine, Spain, and Morocco are the biggest worldwide producers of essential lavender oil. In the last years Bulgaria overtaken on lavender plantations and lavender oil yield (around 100 tons produced yearly) the long-standing leader in this field France (Lesage-Meessen et al. 2015). The main specie grown in Bulgaria is the true lavender (*Lavandula angustifolia* Mill.). Due to the fact that the concentration of essential oil in the plant materials (0.8-1.3% / fresh plant) is relatively low after extraction or distillation of the important biologically active substances large quantities of wastes remain. Throwing simply away or using as compost is among the very often used procedures to eliminate these wastes. But they could also serve as initial materials for recovery of valuable by-products which could be used in the food, cosmetic and perfumery industry. Alternative methods of valorization have also been applied in recent years - fermentation of distilled lavender and biotransformation of terpene compounds into valuable and difficult to chemically synthesize substances (Daramwar et al. 2012; Elguea-Culebras et al. 2016; Marumoto and Miyazawa 2011; Lesage-Meessen et al. 2015; Noma and Asakawa 2010), isolation of substances with strong antioxidant activity – apigenin, rosmarinic acid, luteolin, etc. (Lesage-Meessen et al. 2015; Méndez-Tovar et al. 2015), as well as the use of ethanol extracts from lavandin waste materials for potential antifungal activity (against *Penicillium verrucosum* Dierckx), a common microorganism causing loss in cheese production) (Lesage-Meessen et al. 2015).

Despite of this, very few studies are currently available on the chemical composition and antioxidant capacity of extracts of true lavender (*Lavandula angustifolia* Mill.) waste. Additionally, in the literature are missing data on the chemical composition and antioxidant capacity of wastes after supercritical CO_2 extraction of lavender (*Lavandula angustifolia* Mill.). These observations determined the aim of the present work – to explore the possibility of utilization of spent lavender biomass (obtained by traditional steam distillation and supercritical CO_2 extraction), with an emphasis on obtaining polyphenol rich extracts and to investigate their chemical composition and antioxidant capacity.

Materials and Methods

The lavender wastes were provided by ECOMAAT distillery (Mirkovo, Bulgaria, 2016). The first one was residue after steam distillation of the fresh bio lavender (SD-L) and the second one – residues of supercritical CO_2-extracted bio lavender (CO2-L).

After treatment the SD-L waste was cooled down, inspected for elimination of impurities and dried under vacuum at 50°C. The CO2-L waste was removed from the extraction cylinder and checked for impurities. Both wastes were stored at -18°C until further treatment. All the solvents used were of analytical grade and purchased from local distributors. The 70 % ethanolic extracts from two wastes of *Lavandula angustifolia* (steam distilled: SD-L, and extracted by supercritical CO_2: CO2-L were obtained according to Kratchanova et al. (2008).The total polyphenol content of ethanolic extracts was determined using the method described by Singleton and Rossi (1965). The antioxidant activity by ORAC and HORAC assays was measured as described by Číž et al. (2010). The DPPH and FRAP analysis were performed according to the procedure described in Slavov et al. (2017).

The individual phenolic and flavonoid components were analyzed on Agilent 1220 HPLC system (Agilent Technology, USA), equipped with binary pump and UV-Vis detector. Wavelength of λ= 280 nm was used. Separation was performed using Agilent TC-C18 column (5 μm, 4.6 mm x 250 mm) at 25ºC. Mobile phases constituted of 0.5 % acetic acid (A) and 100% acetonitrile (B) at flow rate 0.8 ml/min. The gradient conditions started with

14% B, between 6 min and 30 linearly increased to 25% B, then to 50% B at 40 min. The standard compounds (gallic acid, 3,4-dihydroxy benzoic acid, chlorogenic acid, caffeic acid, p-coumaric acid, ferulic acid, ellagic acid, catechin, epicatechin, rutin, naringin, myricetin, quercetin, naringenin and kaempherol) were purchased from Sigma-Aldrich (Steinheim, Germany).The individual volatile and non-volatile compounds in the ethanolic extracts were determined according to the following procedures:

1). Non-volatile substances – 0.2 ml ethanolic extract was lyophilized and 50 µL pyridine and 50µLN,O-Bis-(trimethylsilyl)-trifluoroacetamide (BSTFA) were added. The sample was incubated at 70°C for 40 min. For analysis 1.0 µL from the solution was injected on gas chromatograph Agilent GC 7890 with mas-selective detector Agilent MD 5975 and column HP-5ms (30 m with diameter 0.32 mm and 0.25 µm thicknesses). The following temperature regimen was used – initial temperature 100°C (hold for 2 min) then increase to 180°C with 15°C.min^{-1} (hold for 1 min) and increase of the temperature to 300°C with 5°C.min^{-1} (hold for 10 min); injector and detector temperatures – 250°C, helium was used as carrier gas at 1.0 ml.min^{-1}. The scanning range of mass-selective detector was m/z = 50 – 550 in split-split mode (10:1).

2). Volatile substances – 1.0 ml ethanolic extract was extracted with 1.0 ml dichloromethane (triple). The combined organic layers were dried under vacuum at 30°C. To the dry residue 100 µL dichloromethane was added. For analysis 1.0 µL from the solution was injected on gas chromatograph Agilent GC 7890 with mas-selective detector Agilent MD 5975 and column HP-5ms. The following temperature regimen was used – initial temperature was 40°C and then increase to 300°C with 5°C.min^{-1} (hold for 10 min); injector and detector temperatures – 250°C, helium was used as carrier gas at 1.0 ml.min^{-1}. The scanning range of mass-selective detector was m.z^{-1} = 40 – 400 in splitless mode. The individual compounds were identified comparing the retention times and the relative index (RI) with those of standard substances and mas-spectral data from libraries of The Golm Metabolome Database and NIST'08 (National Institute of Standards and Technology, USA).

Statistical analysis. The analyses were run three times, and the data were given as mean values. Statistical significance was detected by analysis of variance (ANOVA, Tukey's test; value of $p<0.05$ indicated statistical difference). The homogeneity of variances assumption was checked by Levene's test.

Results and Discussions

Obtaining of ethanolic extracts from lavender waste. Pretreatment of the plant materials with aqueous-ethanolic solutions is usually applied before extraction of polysaccharides from the raw or waste mass. The aim is to remove some low-molecular substances and secondary metabolites (pigments, sugars, etc.) which will hamper further extraction process. In our case it also aimed at obtaining of extracts rich on polyphenolic substances (Slavov et al., 2016). In previous experiments we have investigated the influence of the ethanol concentration on extractability of polyphenols and subsequent polysaccharide extractions (Slavov et al., 2017). Our findings showed that extraction with 70% ethanol solutions gave the optimum results for possibilities of combined valorization of the waste materials of *Rosa damascena* and for this reason we have decided the treatment of the lavender residues after CO_2 extraction or steam distillation to be performed with 70 % ethanol.

Total phenolic substances, individual phenolics and flavonoids, and antioxidant activity of ethanolic extracts from lavender waste. The obtained 70% ethanolic extracts were subjected to preliminary analysis for their total phenolic substances. The results from the analysis are shown in Table 1.

Table 1. Polyphenols and antioxidant activity of 70% ethanol extracts of lavender wastes

№	Waste material	Total phenolics, mg.g^{-1} DW waste	DPPH, µmol TE. g^{-1} DW waste	FRAP, µmol TE. g^{-1} DW waste	ORAC, µmol TE. g^{-1} DW waste	HORAC, µmol $^-$ GAE.g^{-1} DW waste
1	SD-L	10.75± 0.91^a	355.48± 13.12^a	427.36± 23.54^a	5368.09± 92.61^a	2862.85± 71.22^a
2	CO2-L	7.52± 0.62^b	283.21± 17.04^b	311.29± 18.17^b	3133.74± 101.64^b	1967.62± 69.37^b

Data were expressed as Mean ± SD (n = 3).
$^{a, b}$ - different letters indicated that values of the means in the columns are significantly different ($p^* < 0.05$).

The content of total phenolic compounds in SD-L residue were 25% more than in CO2-L which suggests that the CO_2-extraction extracts better the biologically active substances present in the plant matrix. Since polyphenolic compounds present in the extracts are related to their antioxidative properties, in the subsequent experiments, the antioxidant activity of the ethanolic extracts was investigated employing several methods. It is well known that assaying the antioxidant activity of natural antioxidants, it is recommended use of more than one antioxidant assay for a detailed understanding of the antioxidant properties of substances (Číž et al. 2010). For this reason, several assays expressing various aspects of the antioxidant action of polyphenols (ORAC, HORAC, DPPH and FRAP) were used. The methods employed, cover different aspects of the antioxidant action and give a broader view of the antioxidant potential of lavender wastes. The ORAC method measures the ability of the antioxidant to scavenge peroxyl radicals via hydrogen atom transfer. These radicals are physiologically the most important ones, and the hydrogen atom transfer is the most physiologically relevant mechanism of antioxidant action. The HORAC method measures the metal-chelating activity of antioxidants under the conditions of Fenton-like reaction; hence it indicates the protecting ability of compounds against hydroxyl radical formation. The DPPH test gives information on the radical scavenging capacity of an antioxidant via transfer of a single electron, whereas the FRAP method is an indicator of the metal-reducing capability. The higher concetration of phenolics in SD-L results also in a higher antioxidant activity of the ethanolic extracts – by all the methods used the SD-L antioxidant activity was 25 to 40% higher than the CO2-L extract. Furthermore, having in mind the experiments for antioxidant activity of the ethanolic extracts, the individual phenolic acids and flavonoids were determined. The results from the analysis are presented in Table 2.

Table 2. Phenolic acids and flavonoids in 70% ethanolic extracts

Phenolic acids, mg.g^{-1} DW waste	70 % ethanol extracts	
	SD-L	CO2-L
Neochlorogenic acid	0.558±0.10*a*	0.244±0.08*b*
Caffeic acid	0.331±0.09*a*	0.222±0.04*b*
p-Coumaric acid	1.007±0.19*a*	0.585±0.10*b*
Ferulic acid	0.061±0.05*a*	0.077±0.04*a*
3,4-dihydroxy-benzoic acid	0.306±0.09*a*	0.132±0.08*b*
Gallic acid	0.183±0.04*a*	0.064±0.07*b*
Rosmarinic acid	0.177±0.02*a*	0.063±0.01*b*
TOTAL	2.62±0.18*a*	1.39±0.11*b*
Flavonoids, mg. g^{-1} DW waste		
Quercetin	0.293±0.08*a*	0.294±0.04*a*
Quercetin-3-β-glucoside	0.778±0.09*a*	1.351±0.10*b*
Myricetin	0.217±0.06*a*	0.129±0.07*a*
Kaempferol	0.151±0.04*a*	0.066±0.01*b*
Naringin	0.402±0.02*a*	0.304±0.58*b*
Naringenin	0.034±0.01*a*	0.071±0.01*b*
Catechin	1.274±0.10*a*	0.689±0.10*b*
Epicatechin	0.567±0.09*a*	-
TOTAL, mg.100^{-1} ml	3.721±0.11*a*	2.911±0.12*b*

Data were expressed as Mean ± SD (n = 3).
$^{a, b}$ - different letters indicated that values of the means in the rows are significantly different ($p^* < 0.05$).

Again the results suggested that in the SD-L residue the biologically active substances were less extracted than in the CO2-L waste – the total amount of phenolic acids in SD-L and CO2-L were 2.62±0.18 and 1.39±0.11 mg.g^{-1} DW waste, respectively. Similar trend of the results was observed for the amount of flavonoids present in the extracts of both wastes – 3.72±0.11 mg.g^{-1} DW waste for the SD-L and 2.91±0.12 mg.g^{-1} DW waste for the CO2-L residue. From the phenolic compounds determined in both extracts the highest concentration had p-coumaric acid – 1.007±0.19 and 0.585 mg.g^{-1} DW wastes for SD-L and CO2-L, respectively. The catechin (1.274 mg.g^{-1} DW waste) was the most abundant flavonoid in

SD-L, while the quercetin-3-β-glucoside (1.351±0.10 mg.g^{-1} DW) was found to be the most abundant in the CO2-L waste.

Determination of volatile and non-volatile polar substances in lavender waste ethanolic extracts. In the subsequent analysis by GC-MS were determined the volatile and non-volatile polar compounds present in the 70% ethanolic extracts. The results are presented in Tables 3 and 4. From the group of the non-volatile polar metabolites predominate the organic acids – mainly malic acid (344.20±11.08 and 258.14±14.32 μg.g^{-1} DW extract for SD-L and CO2-L, respectively), linoleic acid (151.84±6.88 and 113.88±5.26 μg.g^{-1} DW extract for SD-L and CO2-L, respectively), gluconic acid (100.77±5.32 and 75.53±4.62 μg.g^{-1} DW extract for SD-L and CO2-L, respectively) and stearic acid (170.44±4.87 and 151.58±3.96 μg.g^{-1} DW extract for SD-L and CO2-L, respectively). Beside these acids, in both waste materials were found in significant amounts α-linolenic acid (an essential ω-3 fatty acid), caffeic and protocatehuic acids which had strong antioxidant activities and contribute significantly to the biological value of the extracts. The major aroma constituents found were linalool (30.68±0.99 and 28.86 % of TIC for SD-L and CO2-L, respectively) and linalyl acetate (25.82±0.85 and 20.97±1.05 % of TIC for SD-L and CO2-L, respectively). Linalool and linalyl acetate contribute significantly to the biological activity of the extracts and hence the wastes and were found to inhibit the growth of *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa* (Jirovetz et al. 2006). Besides linalool showed potent insecticidal, antitumor, anti-inflammatory and antimicrobial action against *Staphylococcus aureus*, *Escherichia coli*, *Yersinia enterocolitica, Pseudomonas aeruginosa* and *Klebsiella pneumoniae*, and it was used in medicine, food and plant protection (Jirovetz et al. 2006) Other minor contributors to the antimicrobial, anti-inflammatory and insecticidal activities found in the 70 % ethanolic extracts were β-caryophyllene, (±)-lavandulyl acetate, lavandulol, ocimene, terpinene-4-ol, limonene, etc (Dorman and Deans 2000; Jirovetz et al. 2006).

The steam distillation process extracts the majority of the essential oil components but significant part remains in the distilled biomass (Tiliacos et al. 2008; Zheljazkov and Astatkie 2012). Summarizing the results for volatile and non-volatile polar metabolites in both waste extracts it could be concluded that in all the cases the SD-L extracts (residue) contain the determined compounds in higher concentrations than in the CO2-L extracts (residues). This also suggests that the CO_2-extraction extracts better all the substances (besides the targeted industrially aroma compounds) from the raw plant material.

Conclusions

The present study focus on two plant wastes from essential oil industry – one obtained from steam distilled lavender (SD-L) and one from subcritical CO_2 extraction of lavender (CO2-L). The lavender wastes showed strong antioxidant capacity with potential beneficial effect on addition in foodstuffs. In all the analysis made (antioxidant activity, total and individual phenolic compounds, content of volatile and non-volatile polar metabolites) the SD-L ethanolic extracts showed the highest results (concentrations) than the CO2-L extract. For the first time lavender residues from CO_2-extraction was investigated for its antioxidant activity, polyphenol composition and aroma metabolites, and comparison with SD-L was performed. In general, the results suggested that the lavender waste were promising source of antioxidants and compounds with potent antimicrobial activity, and have the potential for application in the food industry as cheap biopreservative agent.

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Table 3. Polar non-volatile substances in ethanolic extracts. RI - relative index (Kovats retention index).

Compound	RI	SD-L	CO2-L
		% of TIC	
Glycerol	1266	258.39±10.15 [a]	193.79±8.91 [b]
Phosphoric acid	1278	17.27±1.26 [a]	14.45±0.99 [b]
Succinic acid	1310	59.01±2.36 [a]	44.26±3.11 [b]
Glyceric acid	1339	43.42±4.15 [a]	32.57±2.48 [b]
Fumaric acid	1355	28.23±1.69 [a]	21.18±1.35 [b]
Serine	1362	22.22±0.58 [a]	19.17±0.78 [b]
L-Threonine	1390	23.24±1.14 [a]	17.43±1.36 [b]
Malic acid	1488	344.20±11.08 [a]	258.14±14.32 [b]
Pyroglutamic acid	1512	87.90±2.69 [a]	65.92±3.98 [b]
Salycilic acid	1516	20.94±1.17 [a]	15.75±1.54 [b]
L-Aspartic acid	1531	19.34±0.96 [a]	16.71±0.85 [b]
L-Threonic acid	1528	45.86±2.31 [a]	34.40±2.68 [b]
Vanillic acid	1758	16.50±1.41 [a]	12.37±0.97 [b]
Protocatechuic acid	1813	19.72±1.01 [a]	18.29±1.23 [a]
Quinic acid	1843	12.96±0.64 [a]	9.72±0.95 [b]
Syringic acid	1888	18.50±1.12 [a]	17.62±1.36 [a]
Glucitol	1930	106.57±8.51 [a]	79.18±6.35 [b]
Gluconic acid	1991	100.77±5.32 [a]	75.53±4.62 [b]
Glucaric acid	2013	98.90±2.11 [a]	74.17±3.85 [b]
Myo-Inositol	2090	25.04±2.40 [a]	18.78±2.95 [b]
Stearic acid	2132	170.44±4.87 [a]	151.58±3.96 [b]
Caffeic acid	2140	32.37±2.35 [a]	24.28±2.24 [b]
Linoleic acid	2209	151.84±6.88 [a]	113.88±5.26 [b]
α-Linolenic acid	2217	67.45±1.98 [a]	50.59±1.30 [b]
Stigmasterol	3315	56.86±1.83 [a]	42.64±1.75 [b]
β-Sitosterol	3355	20.81±0.97 [a]	15.61±0.95 [b]

Data were expressed as Mean ± SD (n = 3); nd – not determined.
[a, b] - different letters indicated that values of the means in the columns are significantly different ($p^* < 0.05$).

Table 4. Polar volatile substances in ethanolic extracts. RI - relative index (Kovats retention index); % of TIC - total ion current.

Compound	RI	SD-L	CO2-L
		% of TIC	
α-Pinene	939	0.17±0.02 [a]	0.13±0.03 [a]
Camphene	954	0.14±0.01 [a]	0.11±0.01 [a]
1-Octen-3-ol	979	0.20±0.01 [a]	0.15±0.02 [b]
3-Octanone	984	1.91±0.11 [a]	1.48±0.09 [b]
β-Myrcene	991	0.89±0.05 [a]	0.69±0.10 [b]
β-Pinene	979	0.65±0.09 [a]	0.50±0.06 [a]
3-Octanol	999	0.49±0.04 [a]	0.38±0.02 [b]
p-Cymene	1019	0.16±0.01 [a]	0.13±0.01 [a]
Limonene	1025	1.04±0.11 [a]	0.81±0.15 [a]
Eucalyptol	1031	0.73±0.10 [a]	0.57±0.08 [a]
(Z)-β-Ocimene	1039	2.39±0.25 [a]	1.86±0.15 [b]
(E)-β-Ocimene	1049	2.51±0.21 [a]	1.95±0.22 [b]
γ-Terpinene	1060	0.28±0.02 [a]	0.22±0.03 [a]
Linalool	1097	30.68±0.99 [a]	28.86±0.16 [b]
Camphor	1146	0.11±0.01 [a]	0.09±0.01 [a]
Borneol	1169	0.43±0.04 [a]	0.34±0.05 [a]
Lavandulol	1173	1.11±0.09 [a]	0.87±0.08 [b]
Terpinene-4-ol	1177	1.49±0.12 [a]	1.16±0.12 [b]
Cryptone	1183	0.39±0.02 [a]	0.31±0.03 [a]
α-Terpineol	1189	3.14±0.15 [a]	2.44±0.24 [b]
Geraniol	1253	0.21±0.02 [a]	0.16±0.01 [b]
Linalyl acetate	1257	25.82±0.85 [a]	20.97±1.05 [b]
(±)-Lavandulyl acetate	1290	3.67±0.32 [a]	2.85±0.41 [b]
Neryl acetate	1366	0.70±0.02 [a]	0.55±0.06 [b]
Geranyl acetate	1382	1.36±0.04 [a]	1.06±0.06 [b]
β-Bourbonene	1388	0.15±0.01 [a]	0.12±0.01 [a]
β-Caryophyllene	1419	5.00±0.49 [a]	3.89±0.34 [b]
(E)-b-farnesene	1458	4.98±0.36 [a]	3.87±0.25 [b]
Germacrene D	1482	0.96±0.05 [a]	0.75±0.02 [b]
Caryophyllene oxide	1580	0.23±0.01 [a]	0.18±0.02 [a]
n-Heptadecane	1700	nd	0.67±0.02
Farnesyl alcohol	1725	nd	0.97±0.03
n-Nonadecane	1901	nd	1.48±0.08
10-Heneicosene	2093	nd	1.53±0.10
n-Heneicosane	2100	nd	1.67±0.21
n-Docosane	2200	nd	1.03±0.13
n-Tricosane	2300	nd	0.86±0.09
n-Tetracosane	2400	nd	1.42±0.14
n-Pentacosane	2503	nd	1.17±0.12

Data were expressed as Mean ± SD (n = 3); nd – not determined.
[a, b] - different letters indicated that values of the means in the columns are significantly different ($p^* < 0.05$).

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