Effects of high intensity pulsed electric fields on yield and chemical composition of rose essential oil

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Abstract: High intensity pulsed electric fields (PEF) combined with distillation was used to extract essential oil from rose. Three main parameters, namely electric field intensity, pulse number and distillation times, which can affect the efficiency of extraction, were optimized. Then, in order to demonstrate the feasibility of the method, oil obtained under optimal conditions was compared quantitatively and qualitatively with that obtained by conventional hydro distillation. Considering the extraction yield, the optimal parameters combination was obtained with electric field intensity of 20 kV/cm, pulse number of 8 and distillation time of 2 h. PEF treatment had no significant influence on the quality of essential oils. However, there was an increase in methyl eugenol percentage, which is a disadvantage from qualitative point of view.

Keywords: rose, hydro distillation, pulsed electric fields, PEF, extraction, essential oil
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1 Introduction

Rose is one of the most important crops for floriculture industry. There are about 200 species and more than 18 000 cultivars growing worldwide[1]. Rose has been used in the field of food, medicines, perfumes, cosmetics and aromatherapy for a long time. And is usually used for extraction of oil, which is the most widely used essential oil in perfumery. Rose oil is very expensive because of the labor-intensive production process and the low content of oil in rose blooms, thus, it is known as “liquid gold” in the world market[2]. Some varieties of species of Rosa are of great importance for rose oil production, such as Rosa damascena Mill., Rosa gallica L., Rosa moschata Herrm., Rosa centifolia L. and others[3]. Rosa damascena Mill. is primarily cultivated in Turkey, Bulgaria, Morocco, Iran, India, South Russia, South France, China, South Italy, Libya and the Ukraine in the world[4].

Essential oil can be generally extracted from plant materials by a number of procedures including steam distillation, vacuum distillation, solvent extraction and Supercritical CO_2 extraction[5-7]. The oil content and composition can vary significantly with place of origin, harvest season and weather. Further, the method for obtaining the essential oil plays a key role and determines the quality of the oil[8]. Hydro distillation is the traditional and economical method for rose oil extraction, which is still used now[9]. Although the essential oil

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yield is low, this method is easy to operate. Some attempts have been carried out to improve oil yield and oil quality[10,11]. Solvent extraction can improve oil yield, but it is difficult to obtain solvent-free products. Supercritical CO_2 extraction is limited to apply for the shortcoming of high cost.

High intensity pulsed electric fields (PEF) technology is an emerging field of international food research in recent years. It is based on the application of external electric fields that induce the electroporation of eukaryotic cell membranes enhancing the diffusion of solutes[12]. Recently, it has been discovered that PEF can be widely used to extract active ingredients from natural products. The process does not affect the quality of the extracted products but improves the extraction rates and yields of different active ingredients. PEF, with advantages of non-thermal performance, speediness, efficiency, low power and low pollution, is currently used for non-thermal pasteurization of food and extraction of value components from different natural sources[13-16]. It is a novel and promising method to extract active ingredients.

In the present study, in order to utilize local rose resources and improve oil yield, extraction of essential oil from Rosa of Chanbai Mountain by hydro distillation combined with high intensity PEF was investigated. The effects of the electric field strength, the number of pulse and the distillation time on essential oil yield were evaluated. The oil yield, oil composition and extraction time were compared with those of hydro distillation.

2 Materials and methods

2.1 Plant materials

Flowers of Damask rose were collected early in the morning (5:00-7:00 am) of several days in flowering season (June and July) from the rose gardens located in Changbai Mountain of Jilin Province, China. The flowers were picked at half blooming stage and placed for overnight to remove excessive moisture, then stored at –20°C until needed. Mohamadi et al.[17] reported that petals frozen at low temperatures (–20°C) made it possible to preserve petals for three weeks without any considerable changes in the oil yield and composition.

2.2 PEF treatment system

The PEF treatment system developed at the Department of Biological and Agricultural Engineering of Jilin University was used. It consists of a high voltage pulse generator, an oscillograph, a coaxial liquid material treatment chamber and a pump[18,19].

The generator can generate exponentially decaying bipolar triangle pulse waveforms with the pulse duration of 2 μs. The frequency is adjustable, ranging from 1000 Hz to 3000 Hz. The treatment chamber is composed of two stainless steel electrodes, with an interval of 0.1 cm, a length of 0.15 cm, and a diameter of 0.1 cm. The pulse waveform and input voltage to the treatment can be presented on an oscillograph.

PEF treatment procedure is that sample liquids were pumped to treatment chamber and treated continuously by the PEF suited in the industry[20]. The frequency and flow velocity were adjusted to change electric field strength and the number of pulse. In the experiment, the electric field strength ranged from 10 kV/cm to 30 kV/cm and the number of pulse from 4 to 12, respectively.

The pulse number n is calculated as:

$$n = \frac{2\pi r^2 lf}{q} \tag{1}$$

The electric field strength E is calculated as:

$$E = \frac{V_{PP}}{2l} \tag{2}$$

Treating time is calculated as:

$$T = \frac{Q}{q} \tag{3}$$

where, r is the radius of hole of treatment chamber (0.05 cm); l is the distance between two electrodes of treatment chamber (0.1 cm); f is the frequency, Hz; q is the flow velocity (10 mL/min); E is the electric field intensity, kV/cm; V_{pp} is the input voltage shown on oscillograph, kV; Q is the treatment quantity (630 mL); T is treating time (63 min).

2.3 Experimental procedure

2.3.1 Hydro distillation combined with PEF

A 100 g sample of the rose flowers mixed with 600 mL water was grinded with a colloid mill, and then treated by PEF. The material was processed immediately after PEF treatment, applying water-steam distillation in a Clevenger-type micro-apparatus. The 10% sodium chloride is additive, the effect of this additive was evaluated by Shamspur et al.[21]. As reported, although sodium chloride raised the yield of essential oil (21%), it did not considerably influence the oil composition. The distillation temperature is equal to the boiling point of at atmospheric pressure (100°C).

The effects of the electric field strength (10, 15, 20, 25 and 30 kV/cm), the number of pulse (4, 6, 8, 10 and 12) and the distillation time (0.5, 1, 1.5, 2 and 2.5 h) on essential oil yield were tested. The temperature increased 2°C to 4°C during PEF treatment.

The oil yield was measured and reported as w/w percentage. The essential oil was collected, dried over anhydrous sodium sulfate and weighted, and then stored at 4°C.

2.3.2 Comparison between PEF and conventional methods

Same raw materials of all experiments were used to compare the PEF method with conventional methods.

(1) Hydro distillation

A 100 g sample of the rose flowers mixed with 600 mL water was grinded with a colloid mill, with addition of 10% sodium chloride, and then distilled in a micro-distillation apparatus for 2 h, 3 h, 4 h and 5 h.

(2) Hydro distillation combined with PEF

A 100 g sample of the rose flowers mixed with 600 mL water was grinded with a colloid mill, and then processed with PEF treatment and water-steam distillation at optimal conditions.

2.4 Chemical analysis

The identification of oil components and determination of their relative concentrations were carried out by using gas chromatography/mass spectrometry (GC/MS, model: Shimadzu QP2010). The capillary column was Rxi-5 (30 mm × 0.25 mm, film thickness 0.25 μm). Rose oil samples of 2 μL were diluted in 1 mL of hexane, 1 μL of the resulting solution was injected into a column. The experimental conditions were as follows: the initial oven temperature was 75°C for 2 min and then heated to 210°C with a heating rate of 2.5°C/min, programmed to 240°C/min with a heating rate of 10°C/min and then kept constant for 5 min. The Detector and injector temperatures were set to 260°C and 250°C, respectively; and the carrier gas was helium (99.999%) with a flow rate of 1 mL/min; the volume injected was 1 μL of the rose oil and the split ratio was 5:1. All mass spectra were acquired in electron impact mode with electron energy of 70 eV. The ion source temperature was 200°C and full scan mass spectra was acquired in the mass range within 40-350 amu.

The types of rose essential oil compounds were identified by comparison of their retention index (RI) and mass spectra with those reported in literature and stored in the computer library namely National Institute of Standards and Technology, NIST[22-24]. The relative percentage of every compound was determined with normalization method based on the GC peak areas obtained from MS total ion chromatogram.

2.5 Statistical analysis

All the experiments were repeated for three times, and the mean values were reported. Data were subjected to an analysis of variance (ANOVA) using SPSS 13.0 software package (SPSS Inc., Chicago, Illinois, USA).

3 Results and discussion

3.1 Effect of electric field strength on oil yield

The electric field strength was adjusted by changing voltage (V_{pp}). As shown in Figure 1, the essential oil yield was influenced by electric field strength obviously. When electric field strength increased from 10 kV/cm to 20 kV/cm, the oil yield increased from 0.0783% to 0.103%. However electric field strength continually increased from 20 kV/cm to 30 kV/cm, the oil yield decreased from 0.103% to 0.085%. The phenomenon could be explained that the potential difference between the inside and outside of the cell membrane became larger with electrostatic forces, resulting in the disintegration of organelles and cellular structures[25,26], and then the release of volatile substances might be improved. But the too large electric field intensity may induce negative influence. The similar tendency has been reported previously[27]. In this study, the electric field strength at 20 kV/cm was chosen to extract the essential oil.

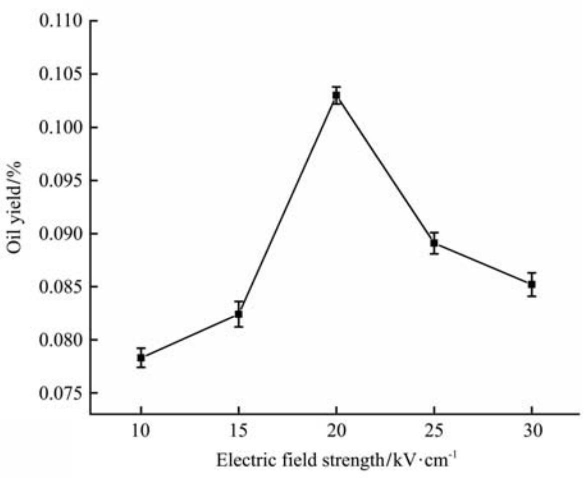

Figure 1 Effect of electric field strength on rose oil yield

3.2 Effect of pulse number on oil yield

The pulse number was changed by adjusting pulse frequency. From Figure 2, it was found that the oil yield was correlated with the pulse number, and the highest value (0.102%) was obtained with the pulse number of 8, decreasing with the number up to 12. According to previous studies of PEF on extraction[28], an increase of pulse number may be shown to cause the increment of diffusion rate within petals and transformation of the cell membrane structure. Therefore, pulse number of 8 was chosen to extract the essential oil.

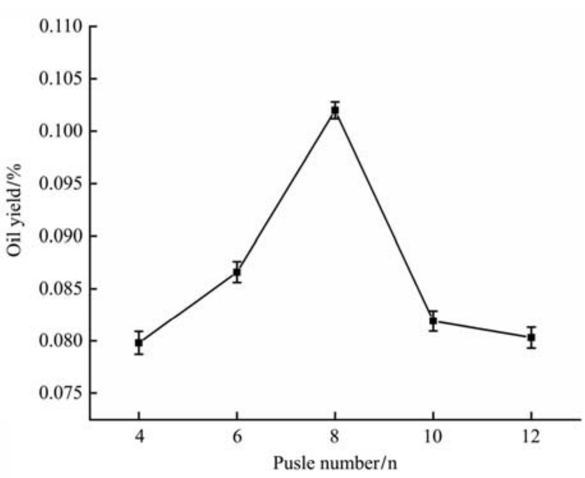

Figure 2 Effect of pulse number on rose oil yield

3.3 Effect of distillation time on oil yield

The effect of distillation time on oil yield was represented in Figure 3. When the distillation time increased from 0.5 h to 2.0 h, the oil yield greatly increased above 64%, and reached the highest value with 0.101%. With regard to the yield, it was observed that a distillation time less than 2 h was not sufficient. While a distillation time of 2.5 h showed a lower yield than the distillation time of 2 h. This might be attributed to oil absorption during the longer distillation. In this context, the distillation time of 2 h could be considered as the best time to extract the essential oil.

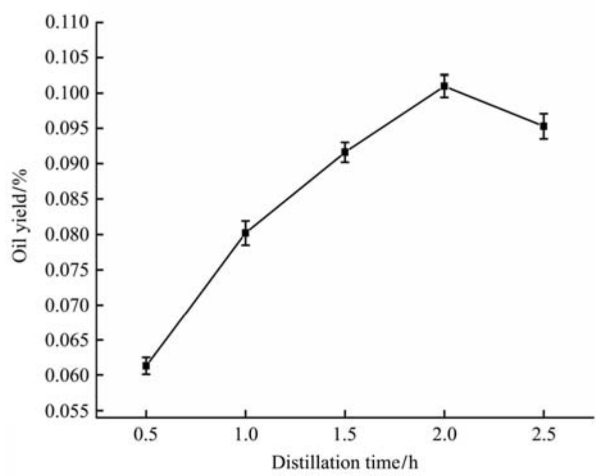

Figure 3 Effect of distillation time on rose oil yield

3.4 Comparison between PEF and conventional methods

To evaluate the efficiency of PEF for the extraction of essential oil from rose flowers, this technique was compared with hydro distillation under optimal conditions. Based on the experimental results, electric field strength of 20 kV/cm, pulse number of 8 and distillation time of 2 h were used. The effect of different methods (Hydro distillation and hydro distillation combined with PEF) on oil yield were illustrated in Figure 4. It can be observed that the essential oil yield increased from distillation time of 2 h to 4 h, and slightly deceased from 4 h to 5 h. The tendency was similar to the report of Baydar which regarded that extending of distillation time up to 3 h could increase the essential oil yield[9]. Obviously, distillation time of 4 h for hydro distillation was the highest with oil yield of 0.0702%. For samples treated by hydro distillation combined with PEF, a yield of 0.105% with increase of 50% was observed compared with only hydro distillation, with distillation time of 2 h. High temperatures for long extraction periods in hydro distillation might cause chemical modification of oil components and loss of the most volatile molecules. Therefore, hydro distillation combined with PEF is an effective method to extract essential oil from rose flowers, due to its higher yield and shorter extraction times.

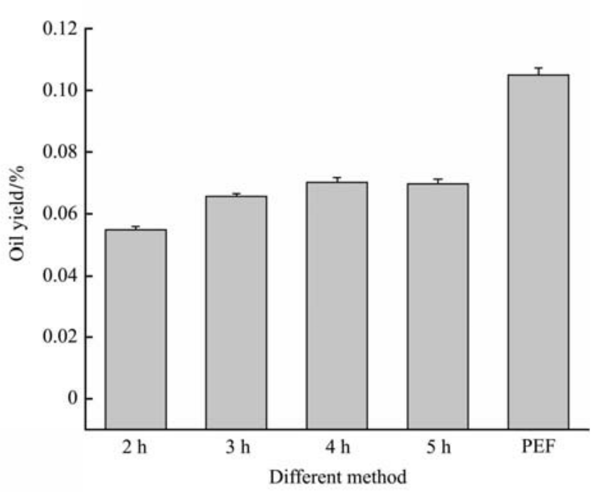

Figure 4 Effect of different methods on rose oil yield

3.5 Chemical analysis

A perusal of data presented in Table 1 reveal that a total of 45 and 50 components were identified for two kind of rose oil extracted by two methods.

The components of rose oil constituted 77.69% and 89.33% of the oil, respectively. There were 36 identical components in the samples obtained by using two different methods. As seen from Table 1, the main components of rose oil extracted by two methods were linalol, citronellol, geranial, neral, rose oxide (cis + tans), eugenol, methyl eugenol and tetracosane. Rose oil is characterized by high percentages of fragrance-bearing components including citronellol, linalool, rose oxide (cis + tans) and eugenol. These components contribute mainly to the perfumery value of rose oil. The majority of components obtained by the two methods were similar, and the important aroma components were also basically the same. It can be concluded that using PEF assistant distillation might not change the components of rose oil.

Many researchers have reported high percentage of the monoterpene alcohols including citronellol, nerol, geraniol, linalool and phenylethyl alcohol in oil rose species, which contribute mainly to the perfumery value of rose oil[3]. In the present study, the content of citronillol was relatively low, and phenylethyl alcohol and geranial were not detected. Depending on ecological factors or genetic variations, damask rose was reported to have these alcohols in low percentages-citronellol of 6% and surprisingly on phenylethyl alcohol[29].

Table 1 Comparison of the chemical composition of rose essential oils obtained by hydro distillation and PEF-assisted distillation

Compound	Hydro distillation/%	PEF-assisted distillation/%
Linalol	4.4	2.02
Heptaldehyde	-	0.71
α-pinene	-	0.11
Phenylethanal	-	0.27
Linalool oxide	-	0.15
Nonaldehyde	-	0.5
cis-Rose oxide	0.85	1.26
trans-Rose oxide	0.27	0.56
Citronellol	2.68	-
Nerol oxide	-	0.17
Geranial	1.31	2.01
Neral	1.82	3.17
2,4-Dodecadienal	0.45	0.62
(E,E)-2,4-Decanedienal	1.24	2.06
Citronellyl acetate	1.38	1.43
Eugenol	3.02	3.98
Germacrene	0.43	0.2
γ-Cadinene	0.49	0.21
Methyl eugenol	7.6	14.49
alpha-cedrene	0.61	0.28
Calamenene	0.34	0.17
γ-Muurolene	2.65	1.83
α-Cubenen	1.59	0.74
β-Guaiene	1.11	0.72
Eremophilene	0.58	0.37
Tridecanone-2	2.59	2.59
Chamigren	2.04	1.34
α-Farnesene	1.81	0.73
Elemicin	-	0.38
β-Cadinene	0.34	-
δ-Cadinene	-	0.31
Tetradecanal	0.37	0.31
Aristrolene	0.2	0.37
γ-Elemene	3.72	2.61
α-Curcumene	4.67	4.41
2-Pentadecanone	2.89	0.81
Isolongifolen-9-one	2.05	9.56
Lanceol	0.65	-
Myristic acid	-	0.37
Geranylgeraniol	0.65	0.38
β-Elemen	0.36	4.68
α-Chamigrene	-	1.23
δ-Elemene	2.06	-
1-Octadecyl acetate	1.39	0.81
n-Hexadecanal	0.65	0.74
a-Amylcinnamyl acetate	2.1	1.99
Farnesylaceton	-	0.65
Palmitic acid	3.88	4.6
n-Octadecylacetate	0.37	-
α-Bisabolool	3.48	4.98
1-Nonadecene	-	0.34
(z)-9-Tricosen	0.28	0.23
Tetracosane	7.58	4.03
n-Icosane	0.79	-
Stearyl aldehyde	0.41	-
1,2-Epoxyoctadecane	-	0.87
Total	77.69	89.33

The overall olfactive quality of rose oil could be impacted by rose oxides and citronellol[2]. The percentages of cis-rose oxide and trans-rose oxide, appropriate content component with high odor unit obtained by the two methods were statistically the same (Table 1). Methyl eugenol is a high value aroma chemical used in cosmetic products and flavoring agents, however, it was not desired above a certain concentration in the essential oils due to side effects on human health[30]. It was found that the relative contents of methyl eugenol increased and eugenol no considerable changed result of different methods.

Higher amounts of hydrocarbons are present in the essential oil of rose flowers extracted by hydro distillation in comparison with PEF-assisted distillation. A low content of hydrocarbons is desirable for high- quality rose oil, so the quality of rose oil might be improved by PEF treatment. The percentage of terpenes compounds is higher in PEF-assisted distillation compared with hydro distillation. This may be because the PEF treatment increased the release of volatile components, and caused components species increasing. In addition, further study may be required to evaluate the effect of different electric field strength and pulse number on composition of rose oil.

4 Conclusions

According to the results obtained, the treatment of rose flowers by PEF before hydro distillation can not only improve maximum oil yield of 50%, but also shorten distillation time for 2 h under the optimal condition with electric field intensity of 20 kV/cm, pulse number of 8 and distillation time of 2 h. The PEF may enhance the release of volatile compounds from blossom tissues by electrostatic forces. As a promising technology, PEF has a broad application space in essential oil extraction of rose and can increase the extraction yield of rose oil.

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