

# Effects of poly(vinyl alcohol) matrix on the release of substances from the fragrance composition<sup>\*)</sup>

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**Abstract:** The chemical composition of two commercially available lavender fragrance oils was determined and compared. The process of concentration by partial evaporation was carried out and its influence on changes in the content of individual components in the analysed mixtures was examined. The effect of poly(vinyl alcohol)-based polymer matrix on the release of volatile substances from oils was tested as well. The presence and chemical composition of the polymer matrix affects the relative content of volatile substances released from the liquid phase containing the tested fragrance composition.

**Keywords:** poly(vinyl alcohol), lavender oil, fragrance oils, hydrogel.

## Wpływ osnowy polimerowej na bazie poli(alkoholu winylowego) na uwalnianie substancji z olejków zapachowych

**Streszczenie:** Określono i porównano skład chemiczny dwóch handlowych olejków o zapachu lawendy. Przeprowadzono proces zatężania kompozycji zapachowych na drodze częściowego odparowania oraz zbadano jego wpływ na zmiany zawartości poszczególnych składników w analizowanych mieszaninach. Zbadano także wpływ osnowy polimerowej na bazie poli(alkoholu winylowego) na uwalnianie substancji z olejków. Stwierdzono, że obecność i skład chemiczny osnowy polimerowej wpływają na względną zawartość substancji lotnych uwalnianych z fazy ciekłej zawierającej badaną kompozycję zapachową.

**Słowa kluczowe:** poli(alkohol winylowy), olejek lawendowy, olejki zapachowe, hydrożel.

Essential oils are an important common group of plant-origin natural raw materials used by man [1]. One of their most common applications is their use as fragrance compositions in household chemical products, such as cleaning agents, fabric softeners as well as air fresheners, among others. Over the years, essential oils have been replaced by less expensive synthetically formulated lipid-based compositions that have a similar but more intense fragrance.

From the chemical point of view, fragrance oils are most frequently complex mixtures of many chemical compounds [2-7]. Individual components of fragrance compositions being chemical substances of very differentiated structure are characterized by various properties, including volatility. Some of them (those more volatile) release into the gas phase easily whereas the others (less volatile) remain in the liquid phase undergoing concentration during the gradual evaporation of the oil. The change of oil chemical composition accompanying the gradual evaporation of individ-

ual components can affect its properties, including the fragrance [8, 9]. It is interesting to study the release profile of individual substances contained in selected oils and the effect of evaporation (concentration) of the oil on the change of the perceptible fragrance. In addition, the presence of certain substances may modify the process of release (evaporation) of individual fragrance ingredients due to their properties (e.g., volatility or viscosity). The objective of the studies was to assess the influence of a solvent (carrier) on the substance release from tested fragrance oils. Besides the oil-water and oil-ethanol systems, the polymer matrices can be applied as carriers. Moreover, poly(vinyl alcohol) (PVA) in the forms of both aqueous solutions and hydrogels, formed due to the crosslinking of PVA macromolecules with tetrahydroxyborate anions, was examined with respect to fragrance profile modification [10, 11]. Hydrogel systems based on crosslinked poly(vinyl alcohol) are biodegradable which is a desirable feature [12, 13] worthy of further research [14-19].

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## EXPERIMENTAL PART

### Materials

The lavender fragrance compositions were obtained from LAW-PS and LAW-MK producers. According to the information in the LAW-PS safety data sheet [17], the oil composition contains dangerous substances whose formulas, names, CAS numbers as well as declared percentage contents are presented in Fig. 1.

Considering the sum percentage of the components declared by LAW-PS, the oil composition also contains other substances which make up for about 76% of the mass.

According to the information given by producer two on the package of the second fragrance composition, i.e., LAW-MK, its main components besides 4-*t*-butylcyclohexyl acetate are coumarin and linalol (Fig. 2).

Polymer matrices (solutions and hydrogels) containing poly(vinyl alcohol) (PVA) were prepared based on the two kinds of PVA. First, with the average molar mass of 130000 g/mol and the average hydrolysis degree of 99% (PVA 130k99) (Sigma-Aldrich, United States). Second, with the average molar mass of 72000 g/mol and the average hydrolysis degree of 87% (PVA 72k87) (Avantor, United States). For preparation of the solutions were used the following: deionized water with specific resistance of 18.2 MΩ·cm (at 25°C) obtained from the water purification system Millipore Elix (Merck, United States), as well as ethanol (EtOH) of the purity ≥ 99.9% (Merck, United States). The PVA-based hydrogel systems were obtained

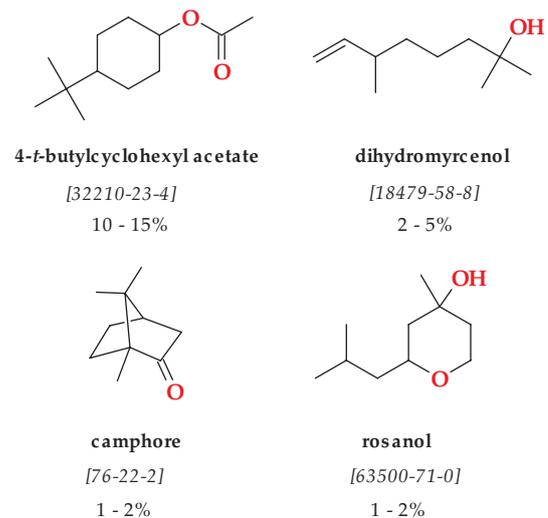


Fig. 1. Main dangerous ingredients of LAW-PS essential oil [15]

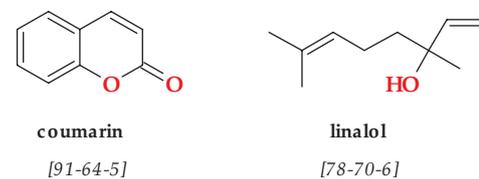


Fig. 2. Chemical structure of selected components of LAW-MK essential oil

from the reaction of polymer macromolecules crosslinking by the tetrahydroxyborate ions  $[B(OH)_4]^-$  formed during dissolution of anhydrous disodium tetraborate (DSTB) (Fluka, United States).

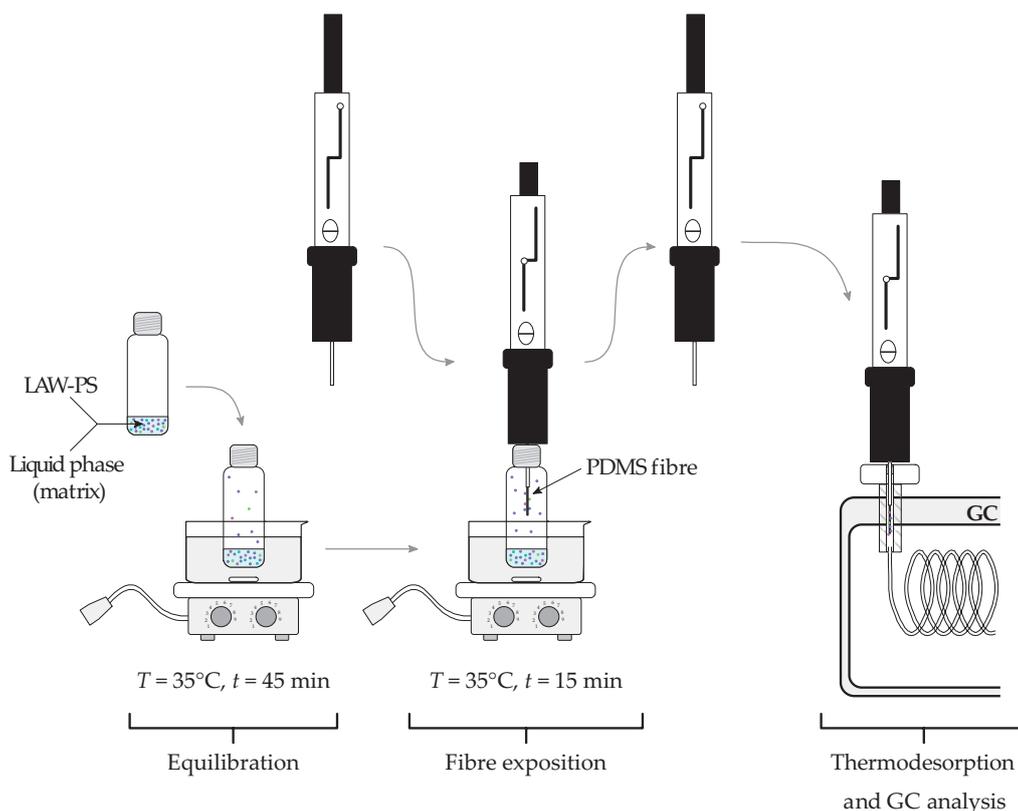


Fig. 3. Scheme of the HS-SPME procedure used in the study of the gas phase composition

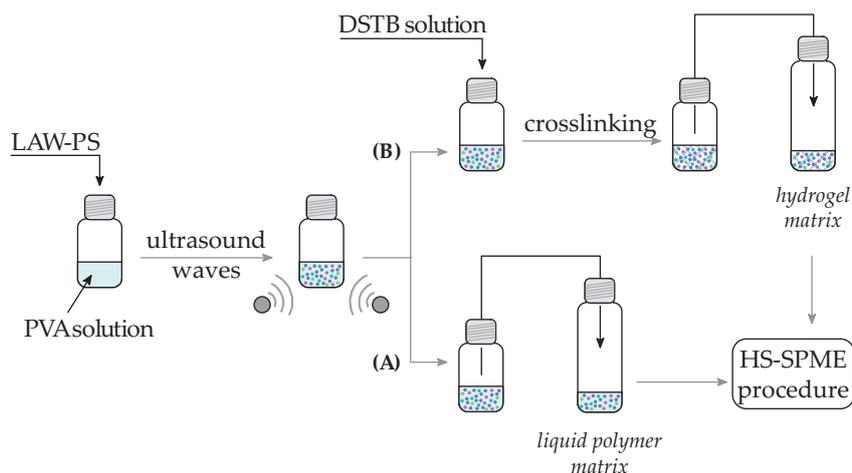


Fig. 4. Procedure for preparing samples of liquid (A) and hydrogel (B) polymer matrix

## Methods

The qualitative and quantitative and compositions of the fragrance substances were analysed using the gas chromatography (GC) method with two different chromatographic systems. Firstly, Shimadzu GC-2010 (Shimadzu, Japan), equipped with split/split less (S/SL) injector, ZB – 5 MSi capillary column ( $L = 30$  m, I.D. = 0.25 mm,  $df = 0.25$   $\mu\text{m}$ ) and flame ionization detector (FID). Secondly, Trace GC ultra DSQ II (Thermo Fisher Scientific, USA) – gas chromatograph (GC) coupled with the mass spectrometer (MS) equipped with the programmed temperature vaporization (PTV) injector and ZB – 5 MSi capillary column ( $L = 30$  m, I.D. = 0.25 mm,  $df = 0.25$   $\mu\text{m}$ ). The liquid samples were injected directly (1  $\mu\text{l}$ ); however, the gas phase composition was determined by the microextraction to the solid phase (SPME) technique from the over surface (headspace) layer (HS) using the 100  $\mu\text{m}$  thick poly(dimethylsiloxane) (PDMS) fiber. The stages of the HS-SPME procedure are presented in Fig. 3.

## Chemical composition analysis

The components of the studied fragrance compositions were identified based on the compatibility of the experimentally obtained (GC-MS) reverse match mass spectrum analysis of the substance peaks available in the NIST17 Mass Spectral Library. While establishing the identity of individual analyte components, the factual possibility of the identified substance presence in the studied sample was considered based on the composition of the lavender oil available in the literature [2-7].

The quantitative estimation of changes in the component contents in the fragrance compositions was made by integration of chromatograms, considering the analytes which concentrations were greater than 0.2%. The percentage content of the identified substances was calculated using the method of interior standardization of peak surface areas on the registered chromatograms.

## Concentration of fragrance components

Concentration (partial evaporation) of the oils was made using the balance-drier MOC-120H (Shimadzu, Japan) coupled with the computer carrying the data acquisition. The samples of fragrance composition (1.0 g) were evaporated by placing them on the glass Petri dish (5 cm diameter) and heating at the atmospheric pressure at 80°C for the time ( $t_{50\%}$ ) indispensable for evaporation of 50% ( $\%m_0$ ) of the initial mass of each sample. From the collected data it was possible to determine the time of evaporation of 10% of the initial sample mass ( $t_{10\%}$ ) as well as the percentage of the mass loss after one hour of the concentration process ( $\%m_{1h}$ ).

Two poly(vinyl alcohol) solutions were prepared by dissolving, firstly, 5 g PVA 130k99 in 250 ml of water, and secondly, 10 g PVA 72k87 in 250 ml of water. 20  $\mu\text{l}$  of the LAW-PS was added to 4 ml of PVA solution. The obtained mixture was sonicated for 5 min. Next, 3 ml of this mixture was placed in a glass vial and the chemical composition of the gas phase was determined using previously described HS-SPME procedure.

Analogous measurements were made for the hydrogel samples obtained for each kind of PVA. To obtain the polymer hydrogel matrix, 20  $\mu\text{l}$  LAW-PS was added to the chosen PVA solution, which was then sonicated. Next, 300  $\mu\text{l}$  aqueous solution of DSTB (in concentration of 10 g per 250 ml) was added to the obtained mixture and was thoroughly mixed. Then, a sample of the obtained hydrogel with a volume of 3 ml was placed in a glass vial with a volume of 20 ml, and after the appropriate time, the gas phase chemical composition was determined using the previously described HS-SPME procedure. The process scheme of the liquid and polymer hydrogel matrix preparation is presented in Fig. 4.

Three independent determinations were made for each sample and the presented quantitative results are the arithmetic mean of the obtained results. Before each successive analysis HS-SPME the PDMS fibre was conditioned for 30 min at 250°C for removal of possible remains from previous study as well as adsorbed impurities.

Table 1. Experimentally determined composition of the LAW-PS

| Peak number | RT, min | RMF | [CAS]      | Molecular formula                              | Name   |
|-------------|---------|-----|------------|--|--|
| 1           | 6.63    | 896 | 80-56-8    | C <sub>10</sub> H <sub>16</sub>                | α-pinene                                     |
| 2           | 7.55    | 847 | 111-13-7   | C <sub>8</sub> H <sub>16</sub> O               | Octan-2-one                                  |
| 3           | 7.81    | 886 | 142-92-7   | C <sub>8</sub> H <sub>16</sub> O <sub>2</sub>  | Hexyl acetate                                |
| 4           | 8.14    | 904 | 470-82-6   | C <sub>10</sub> H <sub>18</sub> O              | Eucalyptol                                   |
| 5           | 8.45    | 893 | 106-62-7   | C <sub>6</sub> H <sub>14</sub> O <sub>3</sub>  | Dipropylene glycol (isomer)                  |
| 6           | 8.51    | 868 | 108-61-2   | C <sub>6</sub> H <sub>14</sub> O <sub>3</sub>  | Dipropylene glycol (isomer)                  |
| 7           | 8.58    | 871 | 18479-58-8 | C <sub>10</sub> H <sub>20</sub> O              | Dihydromyrcenol                              |
| 8           | 8.75    | 858 | 54305-61-2 | C <sub>8</sub> H <sub>18</sub> O <sub>3</sub>  | 3,3'-oxydi(butan-2-ol)                       |
| 9           | 9.04    | 766 | 26532-25-2 | C <sub>10</sub> H <sub>16</sub> O              | (2Z)-(3,3-dimethylcyclohexylidene) ethanol   |
| 10          | 9.51    | 929 | 464-48-2   | C <sub>10</sub> H <sub>16</sub> O              | Camphor                                      |
| 11          | 9.97    | 701 | 107-75-5   | C <sub>10</sub> H <sub>20</sub> O <sub>2</sub> | Hydroxy citronellal                          |
| 12          | 10.16   | 705 | 5332-88-7  | C <sub>10</sub> H <sub>20</sub> O <sub>2</sub> | 4-methylpentan-2-yl butanoate                |
| 13          | 11.35   | 930 | 10411-92-4 | C <sub>12</sub> H <sub>22</sub> O <sub>2</sub> | <i>cis</i> -4-tert-butylcyclohexyl acetate   |
| 14          | 11.69   | 918 | 32210-23-4 | C <sub>12</sub> H <sub>22</sub> O <sub>2</sub> | <i>trans</i> -4-tert-butylcyclohexyl acetate |
| 15          | 12.41   | 887 | 614-60-8   | C <sub>9</sub> H <sub>8</sub> O <sub>3</sub>   | <i>trans</i> -o-coumaric acid                |
| 16          | 13.03   | 801 | –          | C <sub>12</sub> H <sub>14</sub> O <sub>3</sub> | Indan-1,3-diol monopropionate                |
| 17          | 13.58   | 935 | 84-66-2    | C <sub>12</sub> H <sub>14</sub> O <sub>4</sub> | Diethyl phthalate                            |
| 18          | 15.21   | 881 | 110-27-0   | C <sub>17</sub> H <sub>34</sub> O <sub>2</sub> | Isopropyl myristate                          |
| 19          | 15.61   | 888 | 1222-05-5  | C <sub>18</sub> H <sub>26</sub> O              | Galaxolide                                   |

RT = the retention time, RMF = the *reverse match factor*, [CAS] = the CAS (*chemical abstracts service*) number. The blue shading denotes dangerous substances, whose presence in the oil composition is declared by the producer.

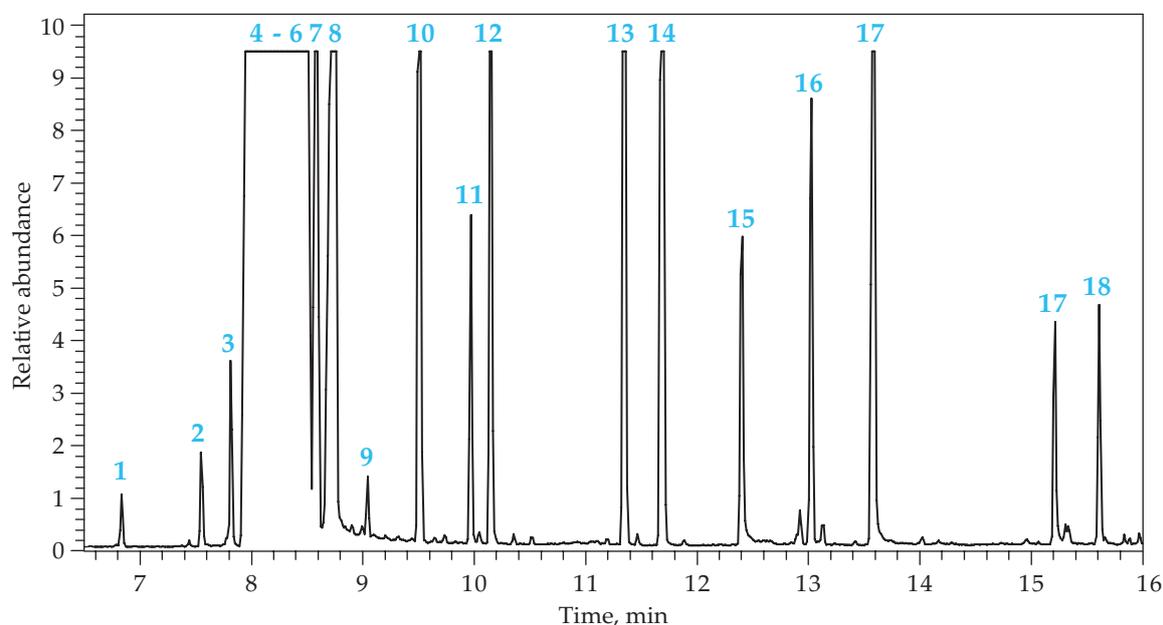


Fig. 5. Chromatogram of LAW-PS essential oil

## RESULTS AND DISCUSSION

The samples of fragrance compositions were subjected to the chromatographic analysis. The obtained results were compared with the qualitative composition declared by the producer (Fig. 1). The chromatogram of

LAW-PS sample is presented in Fig. 5. Table 1 shows the results of qualitative analysis of the sample.

The obtained results indicate that besides the substances declared by the producer one, the LAW-PS contains at least fifteen other components. The values of the parameters characterizing the concentration process of

**Table 2. Comparison of the basic parameters characterizing the process of tested fragrance oils concentrations**

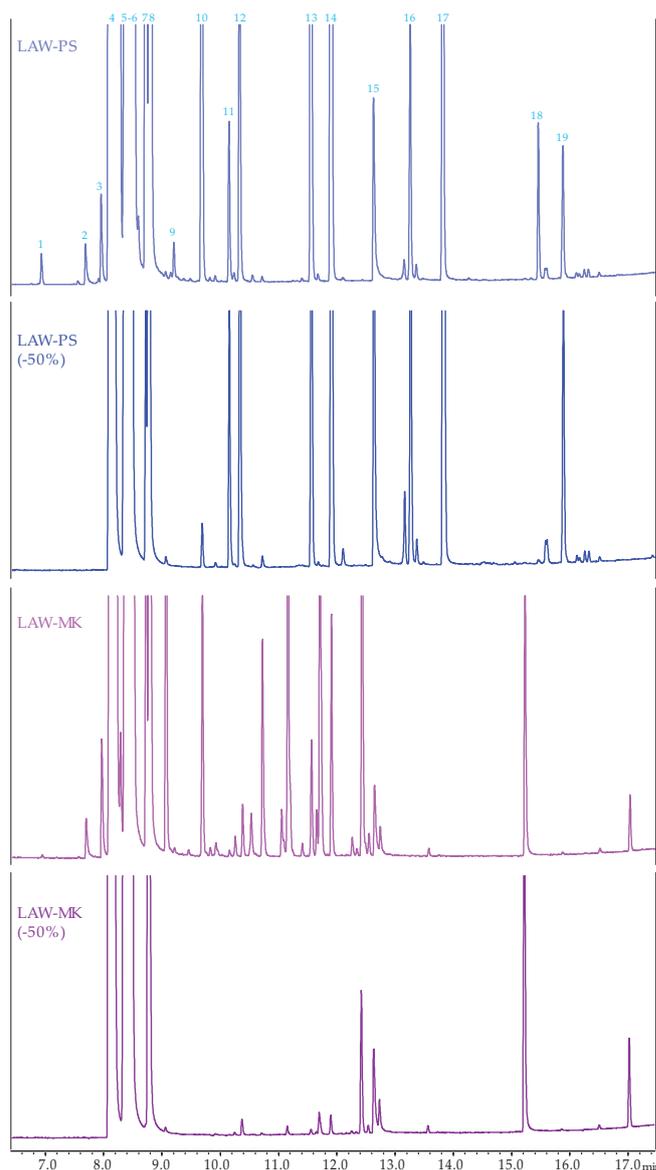
| Fragrance composition | $t_{10\%}$ , min | % <sub>1h</sub> , % | % <sub>m<sub>0</sub></sub> , % | $t_{50\%}$ , min |
|-----------------------|------------------|---------------------|--------------------------------|------------------|
| LAW-PS                | 12.0             | 45.26               | 50.19                          | 69               |
| LAW-MK                | 20.5             | 25.61               | 50.06                          | 138              |

the tested oils by their partial evaporation are presented in Table 2.

As follows from the results presented in Table 2 both lavender oils differ in the chemical composition and the amount of sparingly volatile substances in the LAW-MK is much greater than in the LAW-PS oil. The effect of the concentration process of the fragrance oils on the change of its chemical composition due to the selective evaporation of more volatile components was studied using the chromatographic analysis of the liquid phase of LAW-PS and LAW-MK before and after the concentration process. The obtained chromatograms are presented in Fig. 6.

Although LAW-PS and LAW-MK oils exhibit similar fragrance, the chromatograms confirmed significant differences in their chemical composition. Though some substances, e.g., octan-2-one, hexyl acetate or camphor are components of both products, they differ in the most of other components. The obtained results point out to the change of the oils chemical composition after their concentration. The number of components characterized by greater volatility becomes smaller due to their selective evaporation from the mixture. This results in a decrease of height or complete disappearance of the peaks of these substances in the chromatogram. Comparing the composition of the LAW-PS before and after its partial evaporation one can see a decrease in the camphor content, however the contents of  $\alpha$ -pinene, octan-2-one, hexyl acetate and isopropyl myristate do not exceed their denotation boundary. The differentiated velocity of release of individual fragrance composition components results in the gradual change of the fragrance with time.

As far as the effect of time and temperature on the chemical composition of oils is justified and consistent with the expectations, it can be of interest to study the kinds of matrices in which the oil components are released in the evaporation course. The process of release of LAW-PS oil from the selected matrices was studied to find out a dependence. The chemical composition of the liquid phase (matrix) containing the oil was determined using the HS-SPME technique. There were prepared the systems containing the oil dissolved in ethanol (LAW-PS/E), mixed with water (LAW-PS/W), liquid (LAW-PS/PVA72k87 and LAW-PS/PVA130k99) and hydrogel (LAW-PS/PVA72k87-THB and LAW-PS/PVA 130k99-THB) matrices based on poly(vinyl alcohol). Besides the above-mentioned systems, the analysis of the gas phase composition over the pure oil (LAW-PS) was conducted. The chromatographic analysis was performed and the chemical



**Fig. 6. Chemical composition of LAW-PS and LAW-MK samples after their partial (approx. 50%) evaporation (concentration)**

compositions of selected liquid and gas phases in the equilibrium with them were determined and compared. The selected chromatograms are presented in Fig. 7.

The differences in the chemical composition of the liquid phase of the lavender oil ethanol solution (LAW-PS/E-C) in relation to the phase composition over the solution (LAW-PS/E-G) indicate that application of the readily volatile solvent (ethanol), which enhances vapor pressure over the solution, reduces evaporation of oil components significantly thus modifying the fragrance and reducing its intensity (due to the time range of presented chromatograms, the solvent peak is not visible). The qualitative composition of the gas phase over the pure oil (LAW-PS-G) is like the ethanol composition of fragrance substance solution whereby the relative content of the substance in the gas phase decreases with decreasing volatility. The composition of the gas phase over the

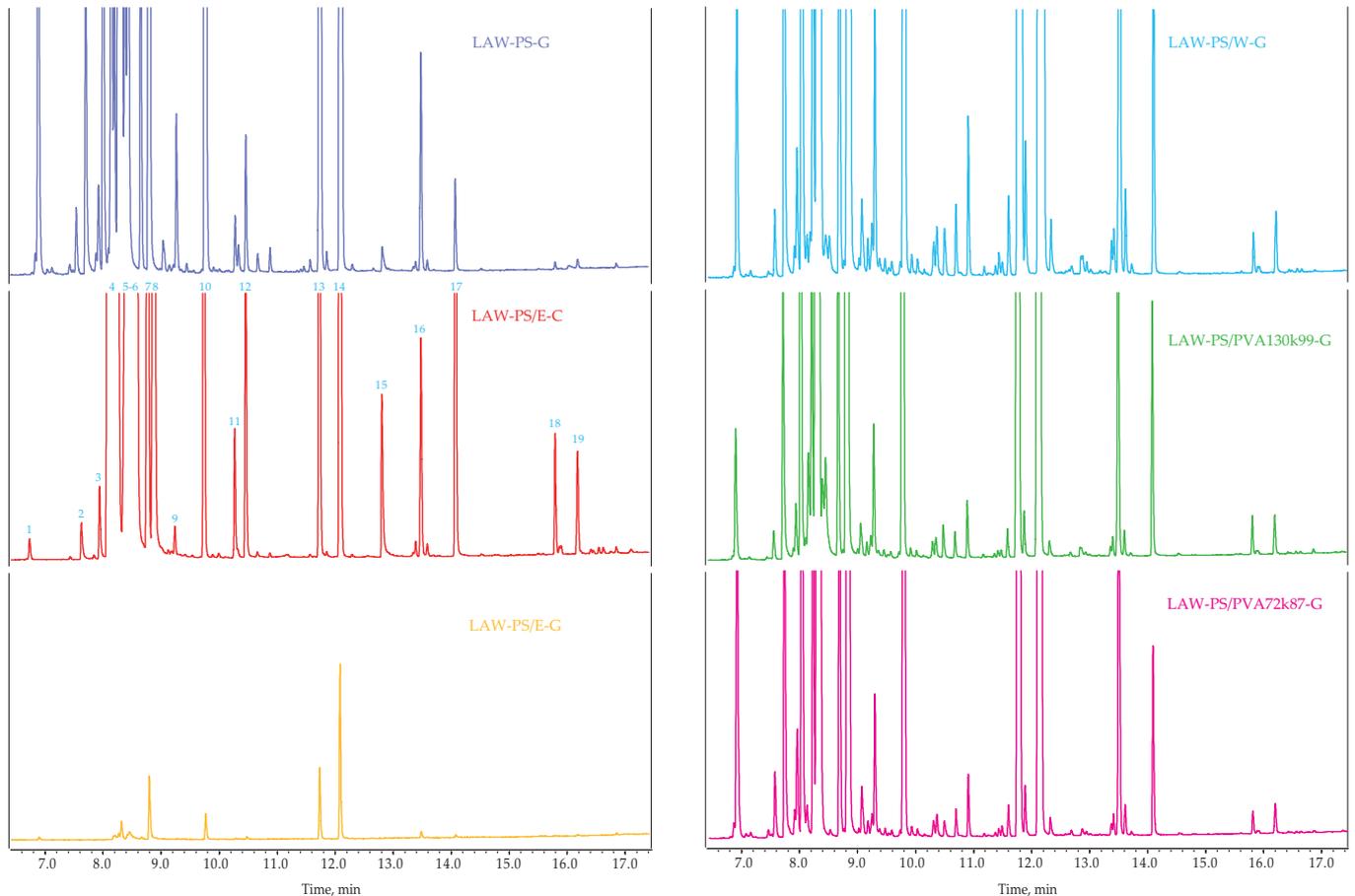


Fig. 7. Chromatograms of selected liquid (C) as well as gas phases (G) that are in equilibrium with the liquid ones

mixture of oil and water (LAW-PS/W-G) turned out to be like the composition of the gas phase over the pure oil.

Compared to ethanol, water is characterized by low volatility in experimental conditions, and with higher polarity, the affinity of some components (especially non-polar ones) to the liquid phase may decrease, thus facilitating their release into the gas phase. The application of liquid polymer matrices (LAW-PS/PVA72k87-G and LAW-PS/PVA130k99-G) did not cause a fabulous change in the composition of the gas phase in relation to the oil-water system. However, the concentration of  $\alpha$ -pinene (RT = 6.04 min) was smaller and that of diethyl phthalate

(RT = 12.98 min) was greater compared to the other components of the over surface phase in the system containing the PVA solution of larger average molecular weight.

The effect of gelation of the liquid polymer matrix on the release of fragrance components was investigated. Therefore, the chemical composition of the gas phase being in equilibrium with the oil-containing polymer hydrogel matrices was determined. The PVA matrix cross-linking was performed using tetrahydroxyborate anions. The selected observed differences are presented in the quantitative aspect in Figs. 8 and 9 as well as Table 3.

Figure 8 shows the comparison of total concentrations of the substances released into the gas phase from the oil-polymer matrix systems. The total surface areas of the peaks registered in the chromatograms were taken as the measure of the analyte content in the gas phase. Determining the total peaks surface areas, only the peaks of the earlier identified substances were considered (1-19). The obtained results can point out to the two tendencies. Firstly, the volatile substance concentrations are greater in the systems based on PVA72k87 than PVA130k99. Secondly, the amount of the components released into the gas phase is smaller than in the case of the hydrogel matrices, i.e. LAW-PS/PVA130k99-THB-G and LAW-PS/PVA72k87-THB-G, compared to the corresponding non-crosslinked matrices (LAW-PS/PVA130k99-G and

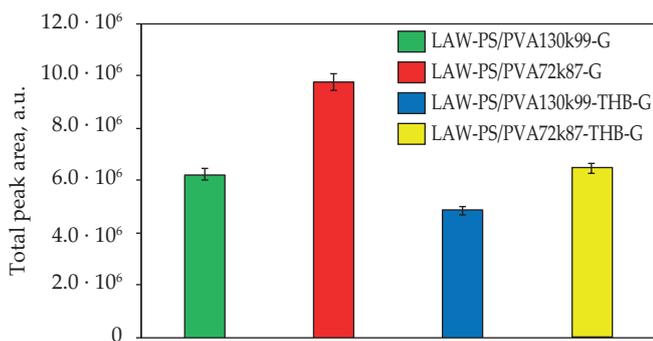


Fig. 8. Total content of volatile substances in the over-surface gaseous phases of LAW-PS/PVA systems

**Table 3. Influence of the polymer matrix type on the quantitative composition of the gaseous phases**

| Substance |                    | Relative content in the gas phase over the surface of the LAW-PS/...-G systems, % |                   |                   |                   |
|-----------|--------------------|---|-------------------|-------------------|-------------------|
| #         | Name               | PVA72k87  | PVA130k99         | PVA72k87-THB      | PVA130k99-THB     |
| 1         | $\alpha$ -pinene   | 0.254 $\pm$ 0.088   | 0.859 $\pm$ 0.128 | 0.643 $\pm$ 0.103 | 1.324 $\pm$ 0.217 |
| 3         | hexyl acetate      | 0.565 $\pm$ 0.094   | 0.591 $\pm$ 0.102 | 0.234 $\pm$ 0.075 | 0.198 $\pm$ 0.063 |
| 4         | eucalyptol         | 1.732 $\pm$ 0.323   | 1.421 $\pm$ 0.299 | 1.954 $\pm$ 0.294 | 1.753 $\pm$ 0.179 |
| 11        | hydroxycitronellal | 0.012 $\pm$ 0.009   | 0.017 $\pm$ 0.011 | 0.010 $\pm$ 0.007 | 0.012 $\pm$ 0.006 |
| 17        | diethyl phthalate  | 0.368 $\pm$ 0.097   | 0.296 $\pm$ 0.099 | 0.054 $\pm$ 0.011 | 0.057 $\pm$ 0.019 |
| 19        | galaxolide         | 0.070 $\pm$ 0.046   | 0.048 $\pm$ 0.032 | 0.095 $\pm$ 0.039 | 0.079 $\pm$ 0.022 |

# = number of the peak in the chromatogram; (mean  $\pm$  std. dev., n = 3)

LAW-PS/PVA72k87-G). The former dependence may result from bigger viscosity of the system containing the poly(vinyl alcohol) solution of smaller average molecular weight which was prepared by dissolution of 10 g polymer and not 5 g as in the case of the PVA130k99 solution. Greater concentration of the polymer causes an increase in the mixture viscosity which makes the diffusion of oil volatile components in the volumetric phase of the matrix difficult and reduces their concentration in the gas phase. The latter effect can be due to the gel consistency of the matrices based on the crosslinked poly(vinyl alcohol). This results in the significant increase of hydrogel systems viscosity and significant reduction of intensity of volatile substance diffusion from the matrix volumetric phase to the gaseous one.

Table 3 presents the comparison of the percentage share of the selected volatile substances in the total amount of the components released into the gas phase from the studied oil-polymer matrix systems. The parameter indicating the content of the substance in the gas phase was established as the surface area of its peak in the chromatogram. The total surface area of registered peaks included only the peaks of earlier identified substances (1-19).

The mentioned results indicate the differentiated quantitative composition of the gas phase depending on the kind of polymer matrix. The results presented in Table 3 point out to the influence of the matrix from which the

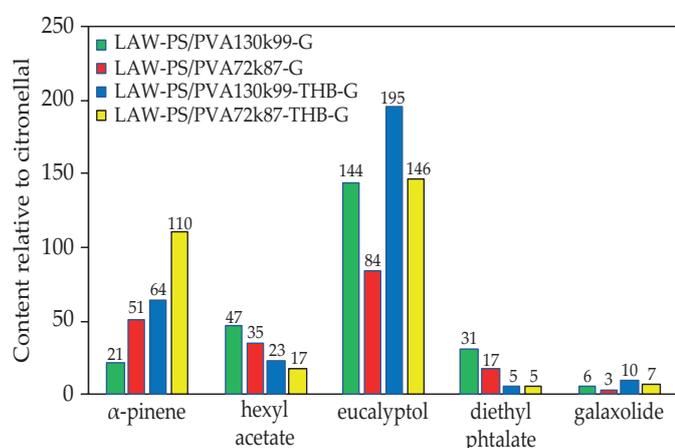
substance was released. The relative contribution of few components in the gas phase decreased over the cross-linked polymer matrices (e.g., hexyl acetate, diethyl phthalate) while that of some components increased (e.g.,  $\alpha$ -pinene, eucalyptol). The contribution of hydroxy citronellal in the relation to the total content of all analysed substances in the gas phase did not depend on the kind of matrix from which release took place.

Figure 9 presents the dependence of the selected substances contents calculated in relation to hydroxy citronellal on the type of polymer matrix. The results presented in the diagram show that hexyl acetate as well as diethyl phthalate exhibited the largest relative content in the gas phase over the non-crosslinked matrix based on poly(vinyl alcohol) of larger molecular weight (i.e. LAW-PS/PVA130k99-G) and decreased after crosslinking of the system (LAW-PS/PVA130k99-THB-G). Similar relationship is visible for the LAW-PS/PVA72k87-G and LAW-PS/PVA72k87-THB-G. Moreover, the concentration of these esters in the gas phase was lower in these systems than in the corresponding matrices containing PVA130k99. The influence of the matrix type on the relative content of the other substances in the gas phase was also evident.

The reason for the influence of the kind of matrix on the concentration of the studied chemical compounds in the gas phase may be the chemical structure of molecules of these substances (e.g., the presence or absence of the specific functional groups) and the resulting properties (e.g., polarity of molecules or volatility). The relative content of fragrance components with a similar chemical structure (e.g., esters) showed a similar dependence on the type of polymer matrix from which they were released. The change of matrix type, including its crosslinking of PVA molecules with tetrahydroxyborate ions, altered the chemical composition, and due to it, kind, and strength of its interactions with the substances contained in the studied fragrance oil.

## CONCLUSIONS

The studies proved that the commercial fragrance compositions are complex mixtures of many chemical substances. Depending on the manufacturer, the qualitative and quantitative composition of lavender oil clearly dif-



**Fig. 9. Influence of polymer matrix type on the relative content of selected substances in the gas phase in relation to the content of hydroxy citronellal**

fers. The volatility of tested oils is the result of the release of liquid phase components into the gas phase. The composition of the gas phase differs from the composition of the liquid phase.

During the gradual evaporation (concentration) of the fragrance oil, both its qualitative and quantitative composition changes. Individual substances contained in the oil may have different volatility, which may lead to selective evaporation of the components (less volatile components are concentrated). The combination of the oil with a polymer matrix based on poly(vinyl alcohol) alters the odour profile, i.e., the relative concentration of individual components in the gas phase - this change depends on the type of matrix.

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