

## VOLATILE COMPOUNDS AND SENSORY PROPERTIES OF COLESLAW MIX PACKAGED IN MODIFIED ATMOSPHERE

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### ABSTRACT

The effect of modified atmosphere and film microperforation on the aroma of a coleslaw mix stored for 12 days at 4°C was detected. Samples were packaged in air and modified atmosphere (5/10/85, 70/30/0% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub>) and sealed with the film with or without microperforations. The application of microperforated film makes it possible to maintain a desirable aroma for 12 days. Use of the unmicroperforated film resulted to increase in allyl isothiocyanate and dimethyl trisulfide concentration. A strong correlation was observed between the following aroma attributes and volatile compounds: sharp - allyl isothiocyanate (R<sup>2</sup>=0.80), dimethyl trisulfide (R<sup>2</sup>=0.83); and carrot - *p*-cymene (R<sup>2</sup>=0.71).

*Keywords:* aroma, cabbage, carrot, minimally processed, modified atmosphere

## 1. INTRODUCTION

In minimal processing, the modified atmosphere packaging (MAP) is frequently used to extend the shelf life of product. In the case of fruits and vegetables, the recommended solution is to expose them to the atmosphere containing 1-5% oxygen and 5-10% carbon dioxide (balanced with nitrogen). A new trend is packaging in superatmospheric oxygen atmospheres (RADZIEJEWSKA-KUBZDELA and CZACZYK, 2016). From literature, it was observed that an increased oxygen content in the atmosphere may inhibit enzymatic discoloration, prevent anaerobic fermentation reaction, influence aerobic and anaerobic microbial growth, reduce decay of the fresh vegetable and also prevent odour losses (DAY, 1996). The studies of CLIFFE-BYRNES *et al.* (2003), and RADZIEJEWSKA-KUBZDELA and BIEGAŃSKA-MARECIK (2009) indicated that aroma is one of the most important factors determining the quality of minimally processed cabbage. Available literature lacks data concerning both the profile of aroma compounds in coleslaw mix and the effect of packaging conditions on the aroma of this product. Research is limited to minimally processed broccoli, carrot, York cabbage or lettuce (CLIFFE-BYRNES *et al.*, 2007; DEZA-DURAND and PETERSEN, 2014; JACOBSSON *et al.*, 2004; LONCHAMP *et al.*, 2009). The profile of aroma compounds may be a significant determinant of quality for such products.

During storage in modified atmosphere, the respiration rate of tissue may lead to a decreased O<sub>2</sub> content and an increase in the CO<sub>2</sub> level inside the package if the gas composition and the permeability of the film is insufficient. This change may result in the formation of fermentative metabolites (such as ethanol, ethyl, acetate, acetaldehyde, methyl acetate and acetone) occurring during anaerobic respiration. Aroma of product from the *Brassicaceae* family is also determined by sulfurous compounds. During the loss of intracellular compartmentalization, enzymes can react with substrates causing strong off-odors. There are mainly products of S-methyl-L-cysteine and S-methylcysteine sulfoxide degradation by the action of cysteine sulfoxide lyase (methanethiol, dimethyl disulfide and dimethyl trisulfide) as well as degradation products of glucosinolates formed as a result of myrosinase activity (AKPOLAT and BARRINGERB, 2015; JANG *et al.*, 2015). Aroma of coleslaw mixes may also be influenced by terpenes originating both from carrot roots and cabbage (VUORINEN *et al.*, 2004; YAHYAA *et al.*, 2015). KJELDSEN *et al.* (2003) reported that during cold storage of carrot, an increase in the contents of terpene compounds to moderate amounts (10-30 ppm) was connected with the carrot aroma, whereas at >35-40 ppm, terpenes produced a harsh and burning turpentine-like flavor. Correlation between aroma and volatile compounds is dependent on the composition of product. Relationship between cabbage and carrot in coleslaw mix was not tested. A better understanding of these relations is needed to facilitate the development of strategies in order to prevent off-odor formation in package.

Our previous studies indicate that the application of modified atmosphere composed of 70/30/0% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub> in packaging of a coleslaw mix with a microperforated barrier film resulted in a good sensory and microbiological quality during the 12 days of storage (RADZIEJEWSKA-KUBZDELA and CZACZYK, 2016). For this reason, an analysis of the profile of volatiles in the coleslaw mix was conducted using the above-mentioned composition in the atmosphere, while for comparison, samples packaged in air and in the atmosphere of 5/10/85% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub> were also tested. In the case of an atmosphere with a 70% oxygen content, a barrier film was also applied in product packaging, as it is frequently recommended for superatmospheric oxygen atmospheres in packaging products of plant origin.

The aim of this study was to determine the effect of modified atmosphere, and film microperforation on the aroma of a coleslaw mix stored for 12 days at 4°C. Correlations

between sensory aroma attributes and contents of volatile compounds in the atmosphere composition were also investigated.

## **2. MATERIALS AND METHODS**

### **2.1. Materials**

Coleslaw mix was produced by mixing shredded white cabbage cv. Galaxy and carrot cv. Perfekcja. The raw materials came from a farm located in western Poland. Prior to technological process, white cabbage and carrot were stored for 1 week at 1°C (this step is not necessary).

### **2.2. Technological process**

White cabbages and carrots were washed in tap water. Thereafter, the outer leaves of white cabbage were removed and the heads were cored using a sharp knife. Carrots were hand-peeled. The vegetables were washed in tap water and dried with absorbent paper. After drying, the vegetables were shredded mechanically, a Nagema HU-1 device (Dresden, Germany) was used for cabbage, while a Robot Coupe CL 50 Ultra device (Vincennes, France) was used for carrot. Shredded cabbage and carrot were mixed at a ratio of 80/20 (w/w). The anti-microbial treatment involved dipping coleslaw for 5 min with agitation in 5 g/L ascorbic acid and 5 g/L citric acid solution. The adherent water after pretreatments was removed using a manual vegetable spinner (Zepter, Viersen, Germany). After that, the shredded material was weighed out (160 g white head cabbage and 40 g carrot) and placed on 205 x 160 x 60 mm polypropylene trays with an oxygen transmission rate of 7-8 cm<sup>3</sup>/m<sup>2</sup>/24 h. The selected atmosphere concentrations % of O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub>: 5/10/85 and 70/30/0, and air atmosphere were introduced into the packages before thermally sealed with a gas packaging device gas mixer (Witt-Gasetechnik, Witten, Germany), Multivac T 200 packaging machine (Wolfertschwenden, Germany). The trays were sealed with an Opalen HB 55 packaging film with oxygen permeability of 35 cm<sup>3</sup>/m<sup>2</sup>/24 h \* atm at 23°C and 85% RH (according to data provided by the film manufacturer) (Bemis, Soignies, Belgium). The film was then microperforated. The microperforations had been made by the Multivac system (Wolfertschwenden, Germany) using one cylinder with 10 needles of 70 µm in diameter (10 microopenings in the film, sealing the tray – 333 holes/m<sup>2</sup> of the film). In the case of modified atmosphere composed of 70/30/0% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub>, the trays were also sealed with the same film but without microperforations.

### **2.3. Gas composition**

Contents of oxygen and carbon dioxide inside the packages were determined using an OXYBABY gas analyzer by Witt-Gasetechnik (Witten, Germany). The results were reported as means of three experimental determinations for separated sample ((RADZIEJEWSKA-KUBZDELA and CZACZYK, 2016).

### **2.4. Analysis of volatile compounds**

The volatile compounds were analyzed after 1, 6, 9 and 12 days of storage at 4°C using solid phase microextraction (SPME). A SPME fiber coated with

divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) was used to collect volatile compounds within each package. To facilitate the headspace-SPME analysis in a semi-quantitative way deuterated (d8) naphthalene (Sigma Aldrich Chemie GmbH, Taufkirchen, Germany) was used as an internal standard (IS). As addition of the internal standard to sampled coleslaw would result in non-uniform distribution of standard, IS adsorption on the fiber was used before sample extraction option. For this purpose, before each coleslaw sampling, SPME fiber was exposed for 5 minutes to IS solution (10  $\mu\text{g}/\text{mL}$ ) in silicon oil. The solute (10 ml) was placed in a 20 ml headspace vial capped with silicon rubber/PTFE membrane. Both IS sampling and subsequent coleslaw sampling was performed at room temperature ( $20^\circ\text{C} \pm 1^\circ\text{C}$ ). The high concentration of IS in the solution compared to fiber capacity allowed for multiple extraction of IS from a single vial. The fiber was exposed to a headspace of the sample for 30 min and after extraction time, the fiber was transferred immediately to an injection port of a gas chromatograph and desorbed for 5 min at  $250^\circ\text{C}$  in a splitless mode. Compound identification was performed using an Agilent 7890A gas chromatograph coupled to a 5975C TAD single quadrupole mass spectrometer (Agilent Technologies, Santa Clara, CA) with a DB-5MS column (25 m  $\times$  0.200 mm  $\times$  0.33  $\mu\text{m}$ , Agilent Technologies, Santa Clara, CA). The carrier gas was helium at a flow rate of 0.8 ml  $\text{min}^{-1}$ , while oven temperature was  $40^\circ\text{C}$  for 1 min, followed by an increase of  $8^\circ\text{C} \text{ min}^{-1}$  to  $220^\circ\text{C}$  and  $20^\circ\text{C} \text{ min}^{-1}$  to  $280^\circ\text{C}$ . Mass spectra were recorded in an electron impact mode (70 eV) in a scan range of  $m/z$  33-333. The mass spectra of volatile compounds were identified tentatively by comparison with spectra of the NIST 05 mass spectral library. Volatile peaks (Total Ion Current) were compared to that of IS and the results provided in ng/g of fresh weight of coleslaw. No correction factors were used.

## 2.5. Sensory evaluation of aroma

Quantitative descriptive analysis was used to characterize the aroma of coleslaw mixes packaged in air and modified atmosphere during 12 days of storage at  $4^\circ\text{C}$ . The panel consisted of 10 members (all employed at the Poznan University of Life Sciences) who were trained in evaluation according to ISO 8586-1. Sensory attributes were selected from literature and from orientation sessions, in accordance with ISO 11035. A total of 8 descriptors were established as the final list. Panelists assessed all descriptive attributes on a 10 cm unstructured line. The results from linear scale were converted into numerical values for data analysis. The 0 value indicated the lowest value intensity and 9 – the highest.

## 2.6. Statistical Analysis

The two-way variance analysis (ANOVA) and Fisher's least significant difference (LSD) were performed and Pearson's correlation coefficients between aroma attributes and contents of volatile compounds were calculated. Statistically significant differences were reported at  $P = 0.05$ . Principal component analysis (PCA) and partial least-squares (PLS) regression analysis were performed using the Statistica version 9.1 computer software (StatSoft Inc., Tulsa, USA).

### 3. RESULTS AND DISCUSSION

#### 3.1. O<sub>2</sub> and CO<sub>2</sub> contents

The results concerning changes in O<sub>2</sub> and CO<sub>2</sub> contents in stored samples are presented in Table 1. After 9 days to the end of the assumed storage time, the oxygen content in samples packaged with microperforated film was uniform and remained at 11.2% to 11.7%. In the case of samples packaged in the atmosphere containing 70/30/0% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub> in the film with no microperforation a superatmospheric oxygen level was maintained throughout storage and after 12 days it was 44.1%.

In samples packaged in an atmosphere with a 30% content of CO<sub>2</sub>, sealed with microperforated film, a significantly ( $P=0.05$ ) higher level of this gas lasted till day 6 of storage. After 9 days of storage, CO<sub>2</sub> contents was observed (from 11.3% to 12.1%) in all tested salad mixes packaged in microperforated film equalization. In turn, in samples packaged in modified atmosphere (70/30/0% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub>) and in film with no microperforation the carbon dioxide content is significantly ( $P=0.05$ ) increased during storage and after 12 days amounted to 46.8%.

**Table 1.** O<sub>2</sub> and CO<sub>2</sub> contents in coleslaw mix stored for 12 days at 4°C.

Content of gas (%)	Storage time (days)	air with film microperforation		5/10/85 % O <sub>2</sub> /CO <sub>2</sub> / N <sub>2</sub> with film microperforation		70/30/0 % O <sub>2</sub> /CO <sub>2</sub> / N <sub>2</sub> with film microperforation		70/30/0 % O <sub>2</sub> /CO <sub>2</sub> / N <sub>2</sub> without film microperforation	
		Mean	Letter	Mean	Letter	Mean	Letter	Mean	Letter
O <sub>2</sub>	1	15.3±0.9	e	8.8±0.9	a	43.1±0.2	f	60.1±0.7	i
	6	12.9±1.0	cd	11.9±0.5	bc	13.9±0.9	d	51.7±0.8	h
	9	11.2±0.2	b	11.7±0.5	bc	11.2±0.6	b	48.3±1.4	g
	12	11.4±0.4	b	11.5±0.6	b	11.5±0.8	b	44.1±1.2	f
CO <sub>2</sub>	1	5.8±0.5	a	10.5±0.6	bc	20.2±1.2	e	30.2±0.4	f
	6	9.2±0.2	b	11.8±0.6	c	14.9±2.1	d	33.5±0.7	g
	9	11.5±0.4	c	11.8±1.1	c	11.3±1.2	c	44.3±1.9	h
	12	11.7±0.8	c	12.1±1.3	c	11.7±0.8	c	46.8±1.0	i

Mean ± standard deviation (n=3) for parameter with different letters are significantly different at  $P = 0.05$ .

Contents of oxygen and carbon dioxide in the atmosphere inside the packaging are influenced both by film permeability and intensity of tissue respiration processes (RADZIEJEWSKA-KUBZDELA and CZACZYK, 2015). The composition of the atmosphere inside the packaging at the application of the barrier film with no microperforation is influenced first of all by respiration processes. A study by JACXSENS *et al.* (2001) showed that packaging of grated celeriac, mushroom slices and shredded chicory endive in a modified atmosphere with a 95% oxygen content in barrier film caused a considerable reduction in oxygen content in the atmosphere of the packaging (to as little as 19.9%, 6.8%, 12.0%, respectively) and an increase in carbon dioxide levels (45.5% for grated celeriac and 47.5% for mushroom slices). In the case of the coleslaw mix, the application of superatmospheric oxygen atmosphere in combination with an elevated content of CO<sub>2</sub> seems to be a factor inhibiting the intensity of respiration processes in finely comminuted tissue. In the tested samples after 12 days of storage the content of CO<sub>2</sub> was approximately 17% higher than after packaging. The effect of the reduced respiration intensity in

butterhead lettuce at the application of a superatmospheric oxygen atmosphere with an elevated (10-20%) content of carbon dioxide was reported by ESCALONA *et al.* (2006).

### 3.2. Volatile compounds

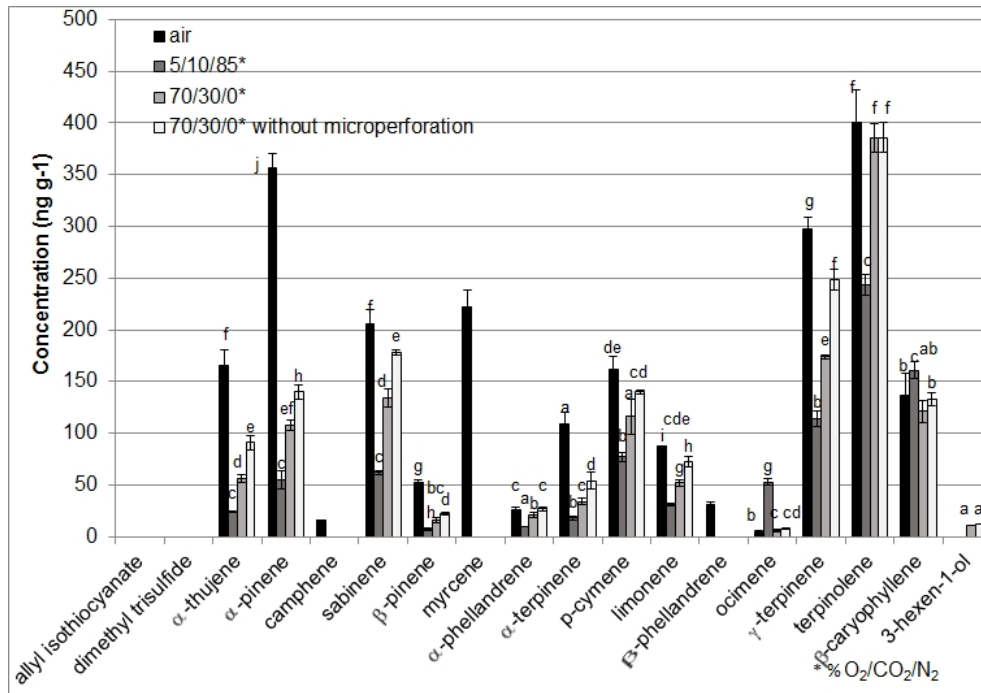
The Figs. 1, 2, 3 and 4 presented the content of volatile compounds in stored coleslaw mixes. Volatile compounds identified in the coleslaw mix included monoterpenes, sesquiterpene, sulfur compounds and alcohol.

The presence of sulfur compounds was recorded only in samples packaged in a modified atmosphere of 70/30/0% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub> in the film with no microperforation. Allyl isothiocyanate was detected after 6, 9 and 12 days of storage, while dimethyl trisulfide was detected after 12 days of storage. The content of allyl isothiocyanate significantly ( $P=0.05$ ) increased during storage. From literature, it was observed that these compounds are responsible for the sharp, sulfury aroma, which in the case of a minimally processed product may be considered undesirable (AKPOLAT and BARRINGERB, 2015). Allyl isothiocyanate is formed as a result of enzymatic (myrosinase) degradation of sinigrin. The accumulation of this compound in the above-mentioned samples may result both from the lack of film microperforation and the high concentration of carbon dioxide inside the packaging. Studies conducted by RADZIEJEWSKA-KUBZDELA and CZACZYK (2015) showed that a high level of CO<sub>2</sub> in the atmosphere may damage cell membrane integrity. In turn, the formation of dimethyl trisulfide is most probably connected with the degradation of S-methylcysteine sulfoxide, under the influence of cysteine sulfoxide lyase (JONES *et al.*, 2004).

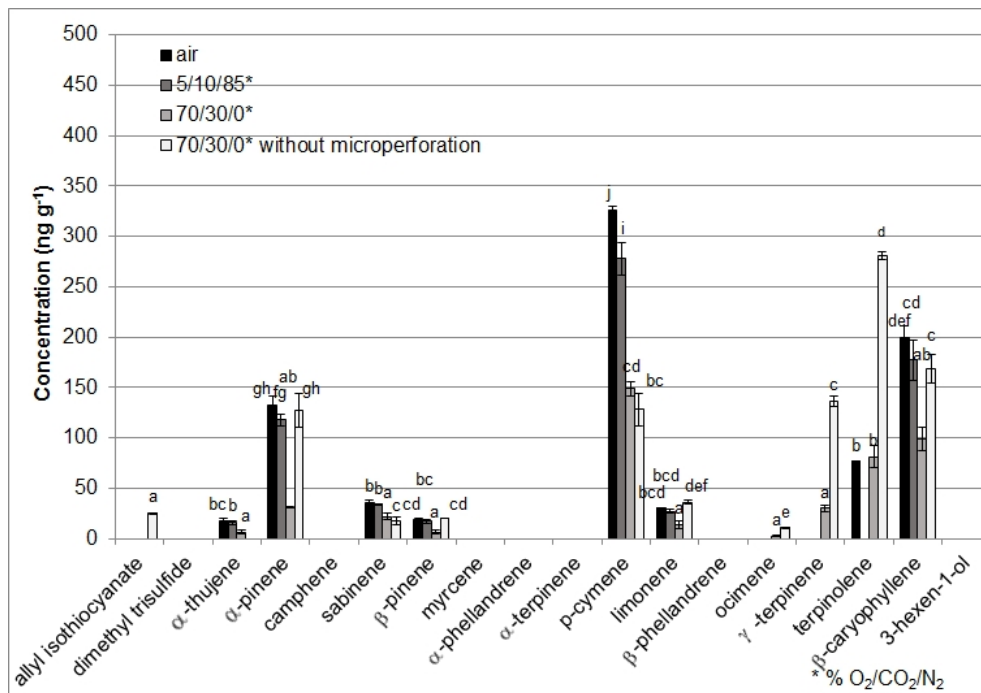
Monoterpenes found in the coleslaw mix may originate both from carrot and cabbage. A study by VUORINEN *et al.* (2004) showed that such compounds as  $\alpha$ -thujene,  $\alpha$ -pinene,  $\beta$ -pinene, sabinene, limonene and  $\gamma$ -terpinene are released as a result of break in continuity of the cabbage tissue. KJELDSEN *et al.* (2003) and CLIFFE-BYRNES *et al.* (2007) indicated the presence of these compounds also in the aroma profile of carrot. Moreover, in that raw material, they identified camphene, myrcene,  $\alpha$ -phellandrene,  $\alpha$ -terpinene, *p*-cymene,  $\beta$ -phellandrene, ocimene and terpinolene and indicated the presence of sesquiterpenes, of which only  $\beta$ -caryophyllene was identified in the coleslaw mix. However, the profile of volatile compounds determined by KJELDSEN *et al.* (2003), and YAHYAA *et al.* (2015) in carrot and cabbage was much broader.

A derivative of the octadecadienoic pathway, 3-hexen-1-ol, was identified only in salads packaged in the atmosphere with a superatmospheric oxygen content after 1 day of storage (Fig. 1). This compound is emitted after mechanical tissue damage and it is labelled as the green leaf aroma.

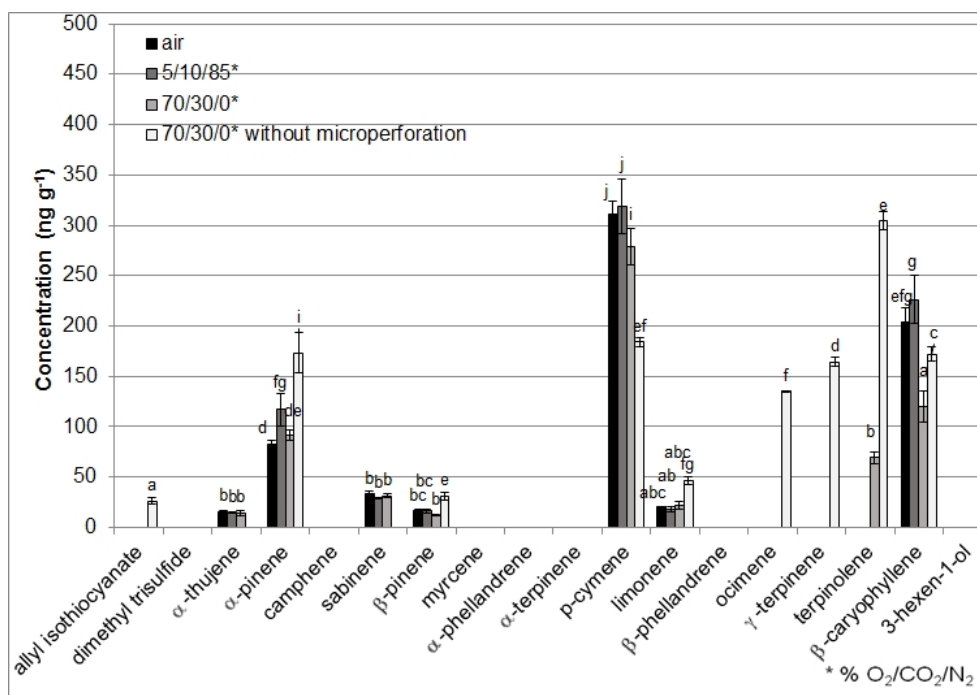
After 1-day storage, salads packaged in air contained camphene, myrcene and  $\beta$ -phellandrene. In turn, in all the tested samples  $\alpha$ -phellandrene and  $\alpha$ -terpinene were detected. In samples sealed with microperforated film  $\alpha$ -thujene, sabinene and  $\beta$ -pinene were detected up till day 9 of storage. However, during storage, a significant ( $P=0.05$ ) decrease in their contents was recorded. In samples packaged in the film with no microperforation the depletion of  $\alpha$ -thujene and sabinene was observed after 6 and 9 days of storage, respectively, while  $\beta$ -pinene was present till the end of the assumed storage period. In the case of the latter compound its content was found to increase significantly ( $P=0.05$ ) during storage. Ocimene,  $\gamma$ -terpinene and terpinolene were found in all the samples only after 1 day of storage.



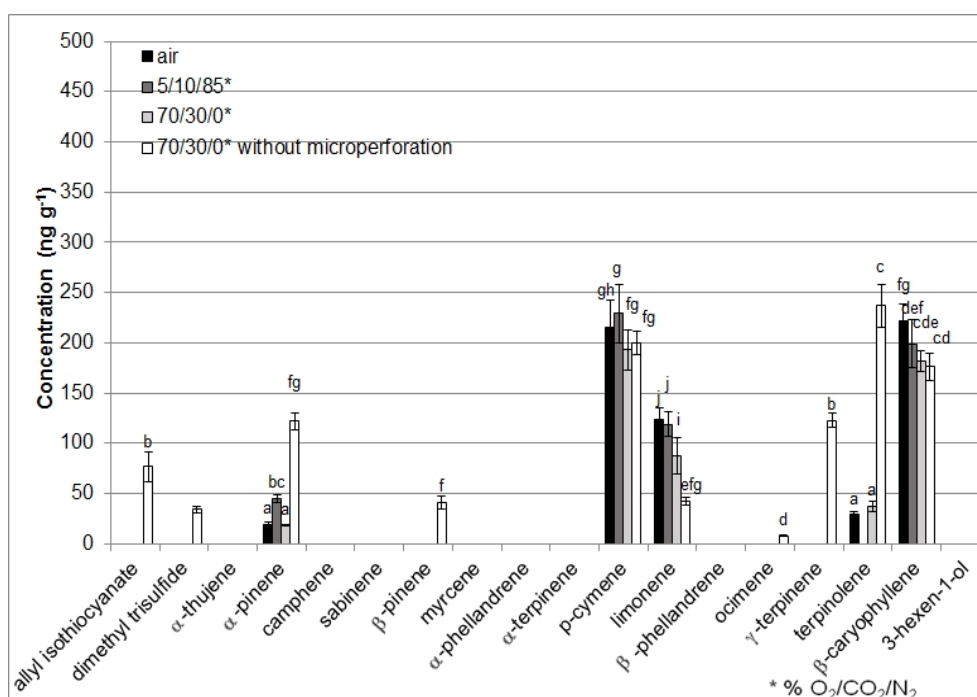
**Figure 1.** Volatile compounds in coleslaw mix after 1 day storage (means (n=3) and standard deviations, different lowercase letters are significantly different ( $P=0.05$ )).



**Figure 2.** Volatile compounds in coleslaw mix after 6 days storage (means (n=3) and standard deviations, different lowercase letters are significantly different ( $P=0.05$ )).



**Figure 3.** Volatile compounds in coleslaw mix after 9 days storage (means (n=3) and standard deviations, different lowercase letters are significantly different ( $P=0.05$ )).



**Figure 4.** Volatile compounds in coleslaw mix after 12 days storage (means (n=3) and standard deviations, different lowercase letters are significantly different ( $P=0.05$ )).

Terpinolene remained present in salads packaged in air and in the modified atmosphere of 70/30/0% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub> up to day 6 of storage. After 9 and 12 days, it was detected only in samples packaged in superatmospheric oxygen atmosphere, while its content was



significantly ( $P=0.05$ ) greater in salad sealed with film and with no microperforation. Ocimene and  $\gamma$ -terpinene after 6 days were still found in samples packaged in the atmosphere of 70/30/0% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub>, while after 9 and 12 days, they were detected only in samples sealed with film without microperforation. Content of ocimene in that sample after 12-day storage was identical to that after 1 day of storage, whereas in the case of  $\gamma$ -terpinene and terpinolene, it was significantly ( $P=0.05$ ) lower. Throughout the entire storage period  $\alpha$ -pinene, *p*-cymene, limonene and  $\beta$ -caryophyllene were recorded in all the tested samples. Their contents were also dominant (Figs. 1, 2, 3, 4). In carrot, KJELDSEN *et al.* (2003) among the main mono- and sesquiterpenes also indicated the presence of  $\alpha$ -pinene, limonene, *p*-cymene and  $\beta$ -caryophyllene, as well as sabinene,  $\beta$ -myrcene,  $\gamma$ -terpinene and  $\alpha$ -humulene, (*E*)- and (*Z*)- $\gamma$ -bisabolene. During storage in the tested samples, a significant ( $P=0.05$ ) decrease in the level of  $\alpha$ -pinene was observed, except for the sample packaged in the atmosphere of 5/10/85% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub>. After 12 days of storage, the highest content of  $\alpha$ -pinene was recorded in the sample packaged using film with no microperforation. In the case of limonene, its contents were found to initially decrease, followed by an increase. After 12 days of storage, the content of limonene in samples packaged with microperforated film was significantly ( $P=0.05$ ) greater than after 1 day, while in the sample sealed using film with no microperforation it was lower. Limonene is a possible oxidation product of chlorophyll in leafy green vegetables, thus its greater concentration in samples packaged in microperforated film may be connected with the depletion of chlorophyll in cabbage leaves (LONCHAMP *et al.*, 2009). In the case of *p*-cymene and  $\beta$ -caryophyllene, their contents were found to increase during storage. After 12 days the content of these compounds in tested samples did not vary significantly ( $P=0.05$ ) (Fig. 1, 2, 3, 4). An increase in contents of mono- and sesquiterpenes during cold storage of carrot was also observed by KJELDSEN *et al.* (2003). The terpenoids may be synthesized in response to physiological stress.  $\beta$ -caryophyllene may be produced by the mevalonate pathway (LONCHAMP *et al.*, 2009) and monoterpenes by the methylerythritol phosphate pathway (BOUVIER *et al.*, 2005).

### 3.3. Sensory analysis

Table 2 illustrated the sensory attributes for stored coleslaw determined by quantitative descriptive analysis.

After 1 day of storage in all the examined samples, aroma attributes defined as carrot and cabbage predominated. After 6 days of storage, a significant ( $P=0.05$ ) deterioration of aroma was observed in samples packaged in film with no microperforation. It was related with an increased intensity of perceived aroma attributes defined as sour and off-odor and a decrease in the intensity of the cabbage aroma. In all the tested samples, an aroma defined as terpene appeared and the green aroma disappeared. After 9 days, a further significant ( $P=0.05$ ) deterioration of aroma was observed in the sample sealed with film without microperforation, which could have been caused mainly by the appearance of the sharp aroma and a greater intensity of terpene aroma. The intensity of cabbage aroma attributes significantly ( $P=0.05$ ) decreased in salads packaged in modified atmosphere using microperforated film. After 12 days of storage, a further significant ( $P=0.05$ ) increase in the intensity of sharp aroma was recorded in coleslaw mixes sealed with film with no microperforation. In those samples in comparison with other samples a greater intensity of sour, terpene and off-odor aroma was recorded at a significantly ( $P=0.05$ ) lesser intensity of cabbage aroma. In salads packaged in microperforated film, the aroma defined as earth

and off-odor was reported. In the case of samples packaged in modified atmosphere a significantly ( $P=0.05$ ) greater intensity of aroma defined as sour was also found.

**Table 2.** Aroma profile of coleslaw mix stored for 12 days at 4°C.

Aroma descriptive attributes	Storage time (days)	air with film microperforation		5/10/85		70/30/0		70/30/0	
				% O <sub>2</sub> / CO <sub>2</sub> / N <sub>2</sub> with film microperforation		% O <sub>2</sub> / CO <sub>2</sub> / N <sub>2</sub> with film microperforation		% O <sub>2</sub> / CO <sub>2</sub> / N <sub>2</sub> without microperforation	
sharp	1	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
	6	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
	9	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	5.3±0.2	b
	12	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	8.0±0.4	c
carrot	1	5.7±0.3	ab	5.2±0.2	a	6.5±0.4	cd	5.9±0.3	b
	6	8.1±0.4	e	7.2±0.4	d	6.3±0.4	bc	5.9±0.2	b
	9	8.3±0.5	e	8.0±0.4	e	6.9±0.4	cd	6.7±0.2	cd
	12	7.3±0.5	d	7.1±0.3	d	6.3±0.4	bc	7.0±0.2	d
sour	1	3.3±0.2	de	3.0±0.3	cd	2.8±0.2	c	4.2±0.3	e
	6	2.2±0.2	b	0.0±0.0	a	2.3±0.2	b	5.8±0.2	g
	9	0.0±0.0	a	3.2±0.4	de	3.4±0.2	d	5.9±0.2	g
	12	0.0±0.0	a	3.9±0.4	e	2.8±0.1	c	5.3±0.2	f
green	1	2.2±0.2	b	2.4±0.3	c	2.5±0.3	c	0.0±0.0	a
	6	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
	9	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
	12	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
earth	1	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
	6	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
	9	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
	12	1.3±0.1	c	1.0±0.1	b	1.1±0.2	c	0.0±0.0	a
terpene	1	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
	6	1.3±0.2	c	1.0±0.1	bc	1.1±0.3	bc	1.2±0.2	bc
	9	1.3±0.2	c	1.0±0.2	bc	1.1±0.3	bc	2.7±0.1	d
	12	0.9±0.2	b	1.3±0.3	c	1.8±0.3	d	4.2±0.1	d
off-odour	1	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a
	6	0.0±0.0	a	0.0±0.0	a	0.0±0.0	a	4.3±0.2	d
	9	0.0±0.0	a	0.0±0.0	a	1.2±0.2	b	4.2±0.3	d
	12	2.2±0.2	c	1.2±0.2	b	1.0±0.2	b	4.3±0.3	d
cabbage	1	6.2±0.4	e	6.3±0.5	e	5.9±0.3	de	8.1±0.5	f
	6	5.3±0.3	c	4.9±0.4	c	5.4±0.3	cd	2.9±0.3	b
	9	2.8±0.3	b	2.9±0.2	b	2.8±0.3	b	3.2±0.3	b
	12	3.2±0.2	b	3.0±0.2	b	3.1±0.3	b	1.8±0.3	a

Mean ± standard deviation (n=3) for parameter with different letters are significantly different at  $P = 0.05$ .

In the tested samples, a strong correlation at  $P \leq 0.05$  was observed between the following aroma attributes and volatile compounds: sharp - allyl isothiocyanate ( $R^2=0.80$ ), sharp - dimethyl trisulfide ( $R^2=0.83$ ) and carrot - *p*-cymene ( $R^2=0.71$ ). Applying the Partial Least Squares techniques, we determined the contribution of volatile compounds to each attribute. In these models, the aroma attributes were Y variables, while the concentration measured by GC-MS were X variables. For each aroma attribute, a proper PLS regression model was established and interpreted using  $R^2$  coefficient. The importance of each volatile for the sensory attributes was determined by variable importance for the projection (VIP) values (Table 3). The  $R^2$  value were satisfactory for sharp, carrot, terpene, off-odour and cabbage. Allyl isothiocyanate and dimethyl trisulfide was the most important contributor to sharp and *p*-cymene for carrot. This confirms dependencies

determined by Pearson's correlation coefficients. Additionally, the dependency between terpene, off-odour, cabbage attributes and volatile compounds was determined (Table 3). Allyl isothiocyanate, dimethyl trisulfide, sabinene and  $\alpha$ -phellandrene were the most important contributor to terpene and ocimene for off-odour. Sabinene and  $\alpha$ -phellandrene, 3-hexen-1-ol,  $\alpha$ -terpinene, allyl isothiocyanate and  $\gamma$ -terpinene were the most important contributor to cabbage. Allyl isothiocyanate is also determined as pungent in Flavor net. KJELDSEN *et al.* (2003) divided terpene compounds present in carrot into carrot ( $\alpha$ -pinene,  $\beta$ -pinene, sabinene,  $\alpha$ -phellandrene, myrcene, *p*-cymene), sweet, citrus, fruity (limonene,  $\gamma$ -terpinene, terpinolene) and terpene-like, spicy, woody aroma compounds ( $\beta$ -caryophyllene). In the tested product only a dependence between *p*-cymene and carrot aroma was observed. Such a correlation may also be indicated by the very low levels of the odor threshold (13 ppb) for *p*-cymene in comparison with  $\alpha$ -pinene (1000 ppb),  $\beta$ -pinene (140 ppb) and sabinene (75 ppb) (KJELDSEN *et al.*, 2003). Moreover, the concentration of *p*-cymene in the tested samples was significantly ( $P=0.05$ ) greater than in the case of other above-mentioned monoterpenes, thus indicating their greater odor activity value. In the case of terpene aroma, sabinene,  $\alpha$ -phellandrene are determined as turpentine in Flavor net. Sabinene and  $\gamma$ -terpinene were also detected as a result of break in continuity of the cabbage tissue as reported by VUORINEN *et al.* (2004)

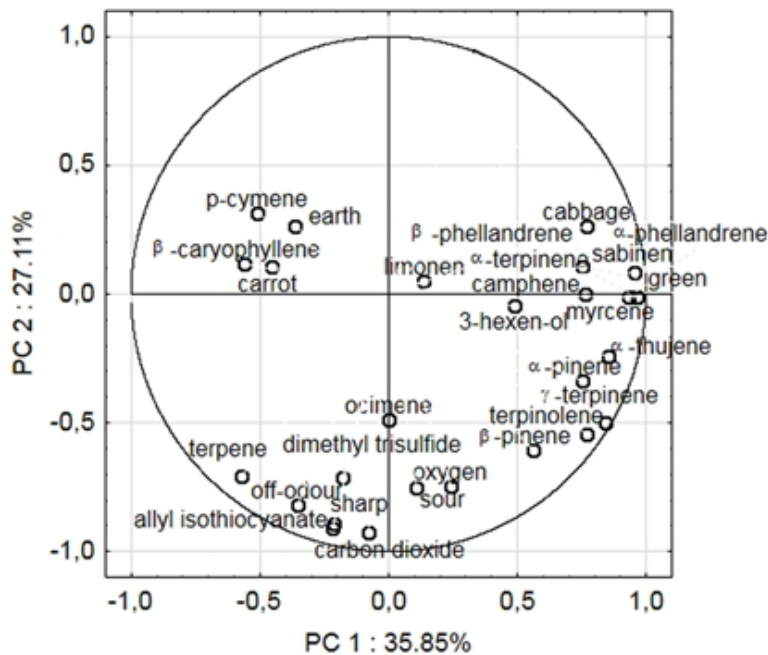
**Table 3.** R<sup>2</sup> for PLS regression model and VIPs affected the aroma attributes.

Attributes	R <sup>2</sup>	Volatiles	VIP
sharp	0.91	allyl isothiocyanate	2.457
		dimethyl trisulfide	2.240
carrot	0.81	<i>p</i> -cymene	2.133
sour	0.54	-	-
green	0.47	-	-
earth	0.45	-	-
terpene	0.84	allyl isothiocyanate	1.870
		dimethyl trisulfide	1.643
		sabinene	1.352
		$\alpha$ -phellandrene	1.237
off-odour	0.77	allyl isothiocyanate	2.207
		dimethyl trisulfide	1.396
		sabinene	1.356
		ocimene	1.289
		$\alpha$ -phellandrene	1.093
cabbage	0.77	sabinene	1.480
		$\alpha$ -phellandrene	1.462
		3-hexen-1-ol	1.316
		$\alpha$ -terpinene	1.190
		allyl isothiocyanate	1.166
		$\gamma$ -terpinene	1.038

### 3.4. PCA

Based on the correlation matrix, 2 principal components were identified, explaining 63% total variability for which correlations with input variables are presented in Fig. 5.

The PC1 was positively correlated with contents of  $\alpha$ -thujene,  $\alpha$ -pinene, camphene, sabinene, myrcene, 3-hexen-1-ol,  $\alpha$ -phellandrene,  $\alpha$ -terpinene, limonene,  $\beta$ -phellandrene,  $\gamma$ -terpinene, terpinolene as well as aroma attributes, that is green and cabbage, while it was negatively correlated with  $\beta$ -caryophyllene, *p*-cymene, carrot and earth aroma. The PC2 was negatively correlated with contents of oxygen and carbon dioxide, allyl isothiocyanate, dimethyl trisulfide,  $\beta$ -pinene, ocimene and aroma attributes, that is sharp, sour, terpene and off-odour (Fig. 5).



**Figure 5.** PCA of volatile compounds, aroma attributes and content O<sub>2</sub> and CO<sub>2</sub>.

Figure 6 presents values of PC1 and PC2 for sample maps. Among the tested samples, the lowest PC2 values were found for salads packaged in film with no microperforation after 6, 9 and 12 days of storage. This was connected with the greatest contents of oxygen, carbon dioxide, allyl isothiocyanate, dimethyl trisulfide, ocimene and  $\beta$ -pinene inside the packaging and aroma defined as sharp, sour, terpene and off-odor. After 12-days of storage the other samples had negative PC1 values, which indicates high contents of  $\beta$ -caryophyllene and *p*-cymene and aroma defined as carrot and to a limited extent earth aroma.

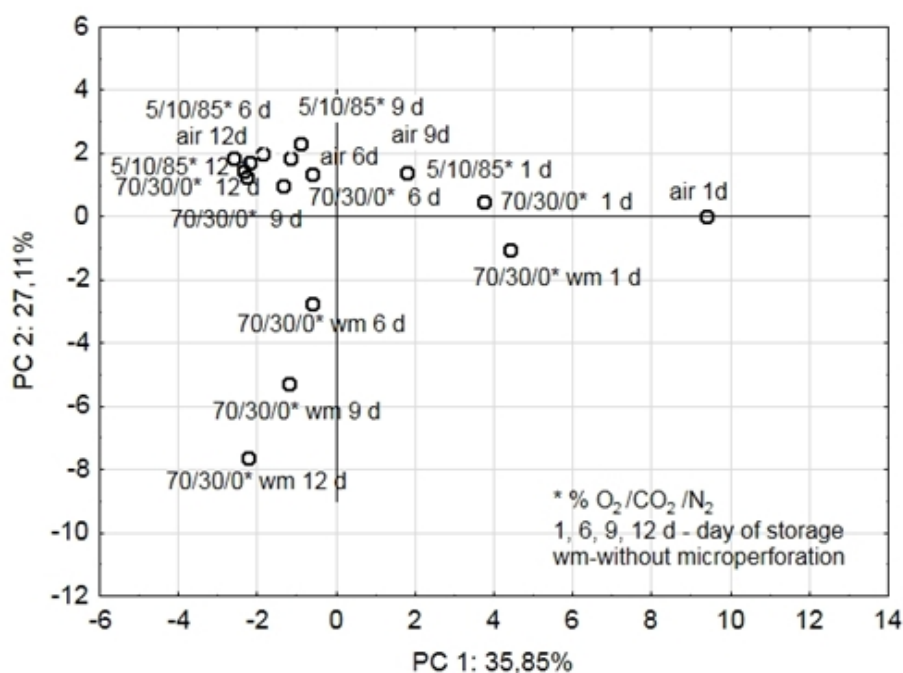


Figure 6. Sample map.

#### 4. CONCLUSIONS

The application of microperforated film in packaging of coleslaw mix makes it possible to maintain the desirable product aroma for 12 days of storage at 4 °C. It also guarantees maintenance of aerobic conditions and does not contribute to the accumulation of CO<sub>2</sub> inside the packaging. Although it may be assumed that in those samples we observed the degradation of chlorophyll found in comminuted cabbage leaves, it may indicate a significantly ( $P=0.05$ ) greater concentration of limonene than in samples packaged in film with no microperforation. The broadest profile of aroma compounds during storage was found in samples sealed in the atmosphere of 70/30/0% O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub>. In the case of packaging the coleslaw mix in the superatmospheric oxygen atmosphere using film with no microperforation, the significant ( $P=0.05$ ) deterioration of aroma in the sensory analysis was the effect of allyl isothiocyanate and dimethyl trisulfide accumulation inside the packaging. Partial least squares regression analysis determined the quantitative contributions of volatile compounds to sharp, carrot, terpene, off-odour and cabbage in coleslaw mix aroma.

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#### REFERENCES

- Akpolat H. and Barringer S.A. 2015. The effect of pH and temperature on cabbage volatiles during storage. *J. Food Sci.* 80:S1878-S1884.
- Bouvier F., Rahier A. and Camara B. 2005. Biogenesis, molecular regulation and function of plant isoprenoids. *Prog. Lipid Res.* 44:357-429.

- Cliffe Byrnes V., Brennan L. and O'Beirne D. 2007. The effects of preparatory procedures and storage temperature on the quality of carrot discs packaged in modified atmospheres. *Int. J. Food Sci. Tech.* 42:482-494.
- Cliffe Byrnes V., Mc Laughlin C. P. and O'Beirne, D. 2003. The effects of packaging film and storage temperature on the quality of a dry coleslaw mix packaged in a modified atmosphere. *Int. J. Food Sci. Tech.* 38:187-199.
- Day, B.P.F., 1996. High oxygen modified atmosphere packaging for fresh prepared produce. *Postharv. News Inform.* 7:31-34.
- Deza-Durand K.M. and Petersen M.A. 2014. Volatile compounds of modified atmosphere packaged cut iceberg lettuce: effect of extremely low O<sub>2</sub>, season, cultivar and storage time. *Food Res. Int.* 62:254-261.
- Escalona V. H., Verlinden B. E., Geysen S. and Nicolai B.M. 2006. Changes in respiration of fresh-cut butterhead lettuce under controlled atmospheres using low and superatmospheric oxygen conditions with different carbon dioxide levels. *Postharvest Biol. Tec.* 39:48-55.
- <http://www.flavornet.org/flavornet.html>.
- ISO 8586-1:1993. Sensory analysis -- General guidance for the selection, training and monitoring of assessors - Part 1: Selected assessors.
- ISO 11035:1994. Sensory analysis - Identification and selection of descriptors for establishing a sensory profile by a multidimensional approach.
- Jacobsson A., Nielsen T., Sjöholm I. and Wendin K. 2004. Influence of packaging material and storage condition on the sensory quality of broccoli. *Food Qual. Prefer.* 15:301-310.
- Jacxsens L., Devlieghere F., Van der Steen C. and Debevere J. 2001. Effect of high oxygen modified atmosphere packaging on microbial growth and sensorial qualities of fresh-cut produce. *Int. J. Food Microbiol.* 71:197-210.
- Jang H.W., Moon J.K. and Shibamoto T. 2015. Analysis and antioxidant activity of extracts from broccoli (*Brassica oleracea* L.) sprouts. *J. Agr. Food Chem.* 63:1169-1174.
- Jones M.G., Hughes J., Tregova A., Milne J., Tomsett A.B. and Collin H.A. 2004. Biosynthesis of the flavour precursors of onion and garlic. *J. Exp. Bot.* 55:1903-1918.
- Kessler A. and Baldwin I.T. 2001. Defensive function of herbivore-induced plant volatile emissions in nature. *Science* 291:2141-2144.
- Kjeldsen F., Christensen L.P. and Edelenbos M. 2003. Changes in volatile compounds of carrots (*Daucus carota* L.) during refrigerated and frozen storage. *J. Agr. Food Chem.* 51:5400-5407.
- Lonchamp J., Barry-Ryan C. and Devereux M. 2009. Identification of volatile quality markers of ready-to-use lettuce and cabbage. *Food Res. Int.* 42:1077-1086.
- Radziejewska-Kubzdela E. and Biegańska-Marecik R. 2009. The effect of composition of modified atmosphere and type of packaging film on sensory quality and physico-chemical properties of coleslaw mix. *EJPAU*, 12.
- Radziejewska-Kubzdela E. and Czaczyk K. 2015. Effect of pretreatment and modified atmosphere packaging on quality of dry coleslaw mix. *Packag. Technol. Sci.* 28:1011-1026.
- Radziejewska-Kubzdela E. and Czaczyk K. 2017. The effect of organic acid pretreatment and modified atmosphere on shelf life of dry coleslaw mix. *J. Food Process. Pres.* 41:1-11.
- Vuorinen T., Nerg A.M., Ibrahim M.A., Reddy G.V.P. and Holopainen J.K. 2004. Emission of *Plutella xylostella*-induced compounds from cabbages grown at elevated CO<sub>2</sub> and orientation behavior of the natural enemies. *Plant Physiol.* 135:1984-1992.
- Yahyaa M., Tholl D., Cormier G., Jensen R., Simon P.W. and Ibdah M. 2015. Identification and characterization of terpene synthases potentially involved in the formation of volatile terpenes in carrot (*Daucus carota* L.) roots. *J. Agr. Food Chem.* 63:4870-4878.

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