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Encapsulation strategy for citrus aroma stabilization using freeze-drying process

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KEY CONTRIBUTION
Core of the microcapsule (*Citrus reticulata* extract) was produced from a byproduct contributing towards the sustainable goals of the food industry. Produced microcapsules exhibited good citrus aroma retention and could be used in the food and cosmetic industry.

ABSTRACT

Commercial juice processing generates a wide range of citrus byproducts including *Citrus reticulata* pomace. Citrus reticulata pomace consists primarily of pectin, cellulose, hemicellulose, and simple sugars. The essential oil present in small amounts contributes to the characteristic citrus aroma. In this research, volatile and semi-volatiles were extracted from citrus pomace (byproduct of mandarin juice production and then encapsulated using a freeze-drying technique. The main goal was to evaluate the efficiency of different coatings such as gum arabic, maltodextrin, and carboxymethylcellulose, to encapsulate citrus aroma. To confirm encapsulation, the microcapsules were disrupted in water. In disrupted microcapsules, a total of 17 monoterpenes, 13 sesquiterpenes, and 15 other compounds were identified, while on the surface of microcapsules, only up to 7 compounds were identified. From 46 aroma compounds identified in disrupted microcapsules, the most abundant ones were limonene, linalool, and α-terpineol.

Introduction

Flavour is one of the most important quality parameters of food products and it is composed of a mixture of volatile compounds. Volatile compounds are organic molecules, usually with a low molecular weight (< 400 Da), which are sensitive to oxygen, heat, and light (Zhang et al., 2015). Volatile compounds are very important secondary metabolites in citrus fruits. They are mainly found in a peel and a pulp and have an important influence on the overall aroma of the fruit. Terpenes, alcohols, aldehydes, acids, and esters are the dominant volatile compounds in citrus (Pichersky et al., 2006; Schwab et al., 2008). Terpenes are the most important volatiles in the peel of citrus fruits (De-Oliveira et al., 1997). In order to prevent volatile compounds from degradation and loss during the processing and storage, it is important to encapsulate them. Encapsulation is a technique by which one material or a mixture of materials is completely covered by another material or the mixture of them. Encapsulated compounds (coated compounds) are usually sensitive compounds such as vitamins, bioactive and volatile compounds unstable under various environmental and processing conditions. The coating material enables a controlled release of the encapsulated compounds under certain conditions (Desai and Park, 2005; Shishir et al., 2018). The coating materials are usually polysaccharides (e.g. maltodextrins, *β*-cyclodextrins, modified starches, etc.), proteins (e.g. whey protein or gelatin), lipids, gums, or polymeric materials. The choice of coating material depends on the nature of the encapsulate, the encapsulation process, and the final use of the encapsulate (de Souza Simões et al., 2017). Due to their great diversity, low cost, and widespread use in the food industry, carbohydrates are most commonly used as coating materials (Dziezak, 1988). The encapsulation is carried out commercially using various methods such as spray drying, spray cooling, extrusion, freeze drying, coacervation, and molecular inclusion. Freeze drying has proved to be the most suitable method for drying thermosensitive compounds such as volatiles. Also, it shows higher retention of volatile compounds and produces powder with rapid solubility (Anandharamakrishnan, 2014; Raja, et al., 2017). The binding and trapping of volatile components depend on the physico-chemical characteristics of the coating material, as well as on the presence of polymers within the complex matrix in the coating material. Also, binding depends on the physico-chemical characteristics of volatile components such as molecular weight, structure, stereochemistry, hydrophobicity, pressure, and solubility (Secouard et al., 2003). Maltodextrin is a carbohydrate obtained by chemical or enzymatic hydrolysis of starch. It is often used for the encapsulation of volatile compounds due to its low cost, low viscosity, neutral taste, and high solubility in water (Sanchez et al., 2011). Gum arabic is one of the common materials used to encapsulate volatile compounds due to its solubility, good retention of volatile compounds and low viscosity. It is also more expensive than some other materials (such as maltodextrin), which limits its use in the food industry (Kenyon, 2005). One of the commonly used coating materials is carboxymethylcellulose (CMC), a cellulose derivate. It is widely used in the food industry as an emulsifier, stabilizer, and, due to its low caloric content, it is also used in low-carb products (Arefian et al., 2020).

There are several studies on the use of carbohydrates, maltodextrin and *β*-cyclodextrin, for the encapsulation of the orange volatile compounds (terpenes), most often in combination with sugars (sucrose, trehalose, lactose, gum arabica, etc.) (Tackenberg et al.; 2014; 2015; Zhu et al., 2014; Farouk et al., 2020). Also, some studies investigated the encapsulation of orange essential oils using a spray-drying process. Maltodextrin, gum arabic, modified starch, and cellulose nanofibrils are known as great polymers for microencapsulation of orange essential oils (Carmona et al., 2013; de Souza at al., 2018; Márquez-Gómez et al., 2018). Sweet orange volatiles, combined with maltodextrin and gelatin in different ratios, showed improved thermal stability and slower volatilization and degradation in freeze-

dried microcapsules (de Araújo et al., 2020). There is a lack of data on the encapsulation of volatile compounds of citrus with maltodextrin, gum arabic, and carboxymethylcellulose using a freeze-drying process. For that purpose, freeze-dried encapsulates of citrus peel and pulp with three different encapsulating agents (maltodextrin, gum arabica, and carboxymethylcellulose) and their combinations were prepared to investigate volatile compounds retention and possible loss in order to evaluate the efficiency of the encapsulation.

Materials and methods

Materials and chemicals

Citrus pomace, which consisted of citrus peel, pulp, membrane residues and seeds, was provided by juice company Regius d.d., Čapljina, Bosnia and Herzegovina. Moisture content of the pomace (80.23 ± 1.65%) was analysed by drying at 105 °C until constant mass was achieved. Water activity of the pomace (0.93 ± 0.02) was measured using LabSwift-aw analyzer (Novasina, Switzerland), while pH value (3.92 ± 0.01) was analyzed by Mettler Toledo pH meter.

Maltodextrin (DE: 16-19), gum arabic and carboxymethylcellulose were purchased from Sigma Aldrich, Saint Louis, MO, USA. All other used chemicals were of analytical grade and obtained from JT Baker, Phillipsburg, USA.

Extraction and encapsulation

Extraction and encapsulation were performed according to the procedure already described (Montero-Calderon et al. 2019; Papoutsis et al.2018). Namely, the extract obtained using ultrasound-assisted extraction with 50% of ethanol was evaporated. Liquid extract was emulsified into the different coating solutions (maltodextrin, gum arabic and carboxymethylcellulose and their combination in same proportions) by stirring at 600 rpm for 60 minutes. Prepared mixtures (coating material + extract) were frozen in the laboratory freezer prior to the encapsulation process at a temperature of − 80 °C for 24 h. Afterwards, samples were freeze-dried under the following conditions: time 48 h, pressure 0.250 mbar and the maximum shelf temperature 18 °C. Following encapsulation procedures, prepared encapsulates were collected and stored in a desiccator until further analysis.

Headspace Solid-Phase Microextraction (HSPME) and Gas Chromatography coupled with Mass Spectrometry Analysis (GC–MS)

Portion of 1 g of microcapsules was put in a 20 mL headspace vial, sealed with a PTFE-silicon septum, and extracted by a manual holder (Supelco, Bellefonte, PA, USA) using DVB/PDMS fiber (Supelco, Bellefonte, PA, USA). The fibers were conditioned according to Supelco instructions. The equilibration of the sample was carried out for 15 min at 60 °C, after which the sample was extracted for 40 min. Thermal desorption of the fiber was performed directly to the GC column for 7 min at 250 °C. A gas chromatograph (7890B Agilent Technologies, Palo Alto, Santa Clara, CA, USA), coupled with a mass-spectrometer detector (model 5977A MSD, Agilent Technologies), was employed to analyze volatile organic compounds isolated from microcapsules. Afterwards, microcapsules were dispersed in 10 ml of water. Water disruption of microcapsule structure (dissolved coating) and HSPME analysis under the same conditions was repeated to determine entrapped flavour compounds.

The separation of volatile organic compounds (VOCs) was performed on an HP-5MS capillary column (30 m × 0.25 mm, 0.25 µm film thickness, 19091 S-433 UI-INT Agilent Technologies, Palo Alto, Santa Clara, CA, USA). The GC–MS analysis conditions and the identification procedure of the compounds were

described previously by Jokić et al. (2022). The compounds were identified by comparison of their mass spectra with the the spectra from the NIST08 and Wiley275 libraries. Retention indices (RIs) were calculated based on the retention times of C_9-C_{20} n-alkanes under the same GC-MS working conditions. The results are expressed as % (peak area of the compound with respect to the overall peak area).

Results and discussion

The two most important characteristics of aroma and flavour microcapsules are encapsulation efficiency and flavour release since these properties may offer a direct assessment of the distribution of aroma constituents both within and on the surface of the microcapsules as well as the evidence of eventual aroma loss during the process (Moran et al., 2014). The intention of the encapsulation process is to obtain more aroma compounds entrapped inside the capsule and less outside on the surface of microcapsules. According to Su et al. (2022), the amount of aroma compounds remaining on the surface of microcapsules should be low enough to avoid aroma evaporation and/or oxidation. The identified components on the surface of the microcapsules mostly belonged to monoterpenes (6), while only one sesquiterpene (δ-cadinene) was detected on the surface of maltodextrin-coated microcapsule. A detailed composition is presented in Table 1. A monoterpene hydrocarbon, limonene, and two monoterpene oxygenated compounds (*α*-terpineol and linalool) were detected on the surface of all studied microcapsules.

Table 1. Compounds identified on the surface of *Citrus reticulata* pomace microcapsules.

No	Compound	Citrus maltodextrin (%)	Citrus gum arabic (%)	Citrus carboxymethylcellulose (%)	Citrus mix coatings (%)
1.	*β*-Myrcene	1.75	-	-	-
2.	Limonene	1.98	0.59	0.97	1.16
3.	*cis*-Linalool oxide	-	0.76	-	-
4.	Linalool	3.08	11.03	1.77	6.16
5.	4-Terpineol	1.06	1.73	-	-
6.	*α*-Terpineol	4.38	9.80	4.39	1.29
7.	*δ*-Cadinene	1.81	-	-	-

RI – retention index, CMC - carboxymethylcellulose, "-" - non-detected

After initial examination (Table 1), microcapsules were disrupted in water. According to Shefer et al., (2003) when dehydrated materials are exposed to water, they rapidly absorb it to replace water molecules that were removed. This process is facilitated by the freeze-dried solid's open structure and high surface area. Disrupted microcapsules were subjected to headspace solid-phase microextraction and gas chromatography coupled with mass spectrometry analysis. The identified components entrapped in the microcapsules belong to different classes of compounds and can be classified into three main groups: monoterpenes 56.72–84.87%, sesquiterpenes 3.19–29.33%, and non-terpene compounds 4.04–5.71%. A detailed composition is presented in Table 2. Cores (compounds inside of the microcapsule) were found to be complex mixtures of compounds belonging to different chemical groups, such as terpenes, carboxylic acids, alcohols, aldehydes, esters, and alkanes. A total of 46 compounds were identified in the microcapsules. Oxygenated monoterpenes were predominant in the microcapsules core and accounted for 38.61–80.84% of the volatile profile, followed by sesquiterpene hydrocarbons 2.6–29.01%, monoterpene hydrocarbons 4.03–18.11%, non-terpenes 4.04–5.71% and oxygenated sesquiterpenes 0.32–0.59%.

Table 2. Compounds identified after disruption of *Citrus reticulata* pomace microcapsules.

No	Compound	RI	Citrus maltodextrin (%)	Citrus gum arabic (%)	Citrus CMC (%)	Citrus Mix coatings (%)
Monoterpene hydrocarbons						
1.	*β*-Myrcene	993	0.15	0.07	0.18	0.03
2.	*α*-Terpinene	1022	0.29	0.16	0.13	0.11
3.	*p*-Cymene	1030	0.34	0.26	0.20	0.15
4.	Limonene	1034	15.10	5.83	9.39	3.27
5.	*γ*-Terpinene	1064	2.23	0.88	1.10	0.47
Monoterpene oxygenated compounds						
6.	*trans*-Linalool oxide	1077	0.06	0.38	0.35	0.53
7.	*cis*-Linalool oxide	1092	0.10	0.42	0.51	0.66
8.	Linalool	1101	23.98	39.67	39.93	42.82
9.	4-Terpineol	1181	2.87	6.22	5.39	6.88
10.	*α*-Terpineol	1193	7.67	18.86	17.24	20.51
11.	*β*-Citronellol	1232	0.96	2.39	1.52	2.03
12.	Carvone	1248	0.21	0.35	0.32	0.42
13.	Geraniol	1258	0.08	0.04	0.03	0.33
14.	Perilla aldehyde	1277	0.32	0.79	0.72	0.88
15.	Thymol	1290	1.19	1.69	1.77	2.23
16.	Perilla alcohol	1299	0.45	0.90	0.65	1.15
17.	Carvacrol	1305	0.72	1.47	1.37	2.40
Sesquiterpene hydrocarbons						
18.	*δ*-Elemene	1340	0.84	0.15	-	-
19.	*β*-Elemene	1393	5.76	1.34	0.74	0.71
20.	*trans*-Caryophyllene	1421	0.43	0.01	0.01	0.01
21.	*α*-Humulene	1455	0.85	0.26	0.21	0.14
22.	*γ*-Selinene	1477	1.30	0.35	0.11	0.05
23.	Germacrene D	1482	1.22	0.18	-	-
24.	*β*-Selinene	1487	3.29	0.53	0.38	0.37
25.	Valencene	1493	2.00	0.27	0.26	0.18
26.	*α*-Selinene	1495	4.05	0.74	0.59	0.45
27.	*α*-Muurolene	1500	0.42	-	-	-
28.	(*E*,*E*)-*α*-Farnesene	1509	5.50	0.63	0.35	0.22
29.	*δ*-Cadinene	1525	3.35	0.87	0.46	0.47
Sesquiterpene oxygenated compounds						
30.	Torreyol	1644	0.32	0.32	0.32	0.59
Other (non-terpene) compounds						
31.	Butanoic acid	< 800	0.08	-	-	-
32.	Ethyl butanoate	804	0.08	-	-	-
33.	Hexanal	805	-	-	0.10	0.09
34.	(*Z*)-Hex-3-en-1-ol	861	0.09	0.18	0.29	0.33
35.	Hexan-1-ol	872	0.05	0.03	0.14	0.21
36.	Octanal	1005	0.10	0.10	0.21	0.18
37.	Phenylacetaldehyde	1050	0.06	0.14	0.06	0.18
38.	Octan-1-ol	1074	0.61	1.88	1.57	1.66
39.	Nonan-1-ol	1175	0.10	0.48	0.22	0.15
40.	Ethyl octanoate	1198	0.12	0.40	0.06	0.04
41.	Dodecane	1200	0.47	0.02	1.59	0.09
42.	Decanal	1207	2.05	1.11	0.57	0.88
43.	Undecanal	1308	0.44	0.01	0.01	0.01
44.	Tetradecane	1400	0.44	0.11	0.87	0.13
45.	Dodecanal	1410	0.21	0.05	0.02	0.09

RI – retention index, - carboxymethylcellulose, "-" – non-detected

Notable differences were observed among different coating materials. According to Trubiano (1998), an ideal aroma-coating material should be water-soluble, have good emulsifying properties and low viscosity as well as low moisture content. In maltodextrin microcapsules, the content of monoterpene hydrocarbons and oxygenated monoterpenes was significantly lower in comparison to other used coatings (gum arabic or CMC). On the contrary, sesquiterpene hydrocarbons were much more efficiently encapsulated using maltodextrin than other coating materials. There are several potential explanations for this observation. One of them is the interaction between core and coating material. Maltodextrin has weaker binding interactions (e.g., hydrogen bonding) with volatile compounds due to its lower molecular weight and lower number of functional groups compared to gum arabic or CMC. This feature limits its ability to retain smaller, more volatile monoterpenes effectively. Another reason could be hydrophobic interactions. Sesquiterpenes are more hydrophobic than monoterpenes and may have a higher affinity for maltodextrin matrices due to hydrophobic interactions. Additionally, considering the general porous and loose structure of the freeze-dried microcapsules in comparison with other encapsulation techniques, it is reasonable to expect a greater loss of aroma compounds (Chen et al., 2013). However, coating choice and combination of coating materials could be beneficial and affect aroma compounds released from the core material. Roberts et al. (1996) evaluated the effect of coating material on dynamic flavour release and found that the application of CMC resulted in a slower release in aroma because of an increase in viscosity and thickness.

Among the most dominant components in the core of the microcapsules were limonene and three monoterpene oxygenated compounds: linalool, 4-terpineol and *α*-terpineol (3.27-15.10%, 23.98-42.82%, 2.87-6.22% and 7.67-20.51%, respectively). Limonene is listed as GRAS (generally recognized as safe) flavouring agent. It has a characteristic citrus aroma, and it is widely used in food, cosmetic and fragrance industries (Sun et al., 2007). The presence of limonene in maltodextrin and carboxymethylcellulose-coated microcapsules was significantly higher than in those coated in gum arabic and mixture of coatings. Opposite was observed for the other three dominant compounds whose presence was lower when maltodextrin was used as coating material. Several encapsulation techniques such as atomization, extrusion, fluidized bed, complex and simple coacervation, spray drying and nano-emulsifying have been evaluated for limonene encapsulation (Ibáñez et al., 2020).

α-Terpineol and 4-terpinelol occurred in high proportions when gum arabic or a mixture of coatings was used. Those compounds, together with carvone, are considered to be off-flavour compounds since they mainly contribute to an unpleasant citrus flavour. They occur as a result of limonene or sometimes linalool degradation via acid-catalyzed reactions (Figure 1).

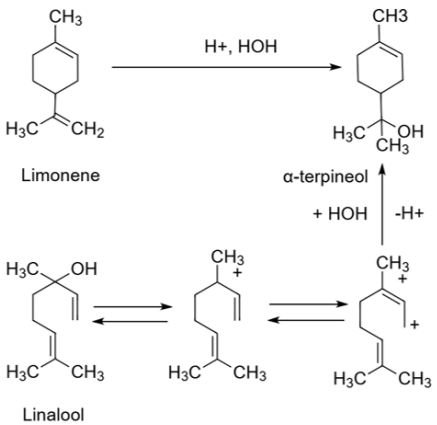

Figure 1. Pathways for linalool and limonene degradation to α-terpineol (adapted according to Haleva -Toledo et al., 1999).

From Table 2 it is evident that with the decrease in limonene proportions, the increase in *α*-terpineol and 4-terpineol occurs. Similar results were obtained by Kaade et al., (2024) where monitoring of limonene degradation was conducted. Also, the same authors found that carvone can occur as the result of limonene degradation. However, in our study this trend was less marked since proportions of carvone were low (from 0.21-0.42%). Several authors reported that coating materials have stabilizing effect on the degradation of limonene (Pérez-López et al., 2006; Sabik et al.,2014; Kaade et al., 2024). Among other used coatings, CMC proved to be the second best coating material and it yielded higher retention of limonene with lower retention of undesirable compounds. Generally, CMC causes a higher viscosity of the coating-extract mixture. As a result, a surface appears that is negatively charged, but has an increased thickness and a higher viscosity. This type of microcapsule surface can decrease acid-catalyzed hydration and dehydration reactions (such as limonene degradation) and prevent the release of deterioration compounds (Kaade et al., 2022). That is also evident from Table 1, where CMC containing coating did not have off-flavour compounds on the surface. Beside core-coating interactions, the formation of an inclusion complex and hydrophobic and polar interactions for the case of maltodextrin and changes in viscosity for CMC (Buljeta et al., 2021), other processing factors can affect aroma retention. Chen et al., (2013) reported that the major disadvantage of freeze drying as an encapsulation technique is the durability of the process. The encapsulated volatile compounds could be released from the core through porous shelsl during ice crystal removing. In this study, the mechanism of aroma retention, beside coating choice, is probably affected by volatile diffusion through the matrix, followed by the evaporation into the environment, since the percentage of limonene varied among different coatings. However, there are several more factors affecting the aroma retention, including the core compounds properties. Yaman et al., (2023) found that volatiles with high molecular weight, low vapour pressure, such as linalool, have better retention in the carbohydrate-based matrix.

Beside terpenes, several components were identified within the group of non-terpenes, including carboxylic acids, alcohols, aldehydes, esters, and alkanes. Within this group, octan-1-ol was the most abundant compound (0.61-1.88%). According to Choi et al., (2001) gas chromatography, coupled with olfactometry analysis, octan-1-ol is the key compound (most odour-active) of Hyuganatsu citrus aroma. They described this aroma as fresh and fruity. The sensory properties of this aroma are important, because the boiling point of the octan-1-ol is similar to limonene as well as myrcene (Kvittingen et al., 2021). When limonene is separated, using hydrodistillation, those compounds (octan-1-ol and myrcene) are usually co-distillated.

Conclusions

This study investigated a sustainable approach for citrus aroma stabilization through waste valorization, green extraction and freeze-drying encapsulation process. The volatile organic compounds profiling revealed the presence of only 7 compounds on the surface and 46 of various compounds in the core, confirming the efficiency of the encapsulation process. Headspace profiling revealed the presence of oxygenated monoterpenes as the most represented group (38.61–80.84%) followed by sesquiterpene hydrocarbons (2.6–29.01%) and monoterpene hydrocarbons (4.03–18.11%), while non-terpenes (4.04–5.71%) and oxygenated sesquiterpenes (0.32–0.59%) were present in small amounts. Among detected compounds, limonene, linalool, 4-terpineol and α-terpineol were dominant. Maltodextrin microcapsules had the highest content of limonene and lower content of off-flavour compounds (4-terpineol and α-terpineol. A notable limitation of this study is that, although the encapsulation of flavour compounds was thoroughly evaluated, the stability of these encapsulated compounds during

extended storage periods was not assessed. Further research can be focused on optimizing the process parameters with the concentration of the coating material used for encapsulation, and evaluating favourable controlled release and unfavourable release during the storage.

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