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# Influence of Carriers on the Functional Properties of Spray-Dried Flavors During Storage

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ENCAPSULATION is a process of protecting functional ingredients like flavors against physical and chemical changes that may occur, especially during storage. In this study, the effect of selected carriers, namely maltodextrin (MD), gum Arabic (GA), and sodium caseinate (SC), was investigated on the functional properties of aroma compounds during storage. Spray-dried aroma compounds (linalool, citral, Orange oil, allylcaproate, and isoamylacetate) were formulated using GA, MD, and SC as carriers with different concentrations then stored in desiccators at room temperature for one year. In addition to moisture content, morphology and extent of non-enzymatic browning (NEB) of stored spray-dried aroma were examined using a scanning electron microscope (SEM) and chromameter. Flavor retention was evaluated using gas chromatography-mass spectrometry (GC-MS). After storage microcapsules for 12 months, the water content of the microcapsules of spray-dried flavorings is ranged between 2.96 to 5.97%. SEM analysis showed little cracks, porosity, or breaks which negatively affected the retention of the flavoring. The spray-dried citral browning recorded the highest intensity, followed by Orange oil, isoamyl acetate, linalool, and allylcaproate, respectively. Linalool and isoamyl acetate were the most retained aroma identified by GC-MS compared to the other investigated stored spray-dried flavors.Better retention overtime was achieved when GA was used in higher content than MD at constant SC. Chemical and physical analysis of the spray-dried microcapsules after storage revealed the possibility of using spray-dried flavors in the food matrix for a long time without affecting the product quality.

**Keywords:** Encapsulation, Flavoring, GC-MS.

## Introduction

Encapsulation is an effective method applied to minimize the negative effect of environmental interactions with active components like flavorings of foods or pharmaceuticals during storage (Poncelet, 2006). The encapsulation could be described as a process of coating active and functional ingredients with biopolymers, e.g., modified starch, pectins, gum Arabic, alginates, and caseinates (Gharsallaoui et al., 2007 and Shahidi & Han, 1993).

Among the different available encapsulation techniques, Spray-drying represents an economic low-cost microencapsulation technology commonly used on an industrial scale with relatively simple, continuous operation and inexpensive, compared to other techniques (Bakry, 2015).

Many authors studied the effect of coating materials and microencapsulation on essential oils or some flavorings during storage; however, neither the effect of the chemical structure of

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aroma compounds nor the nature of biopolymers and their mix were studied before. For example, Kausadikar et al. (2015) used carbohydrates, gum Arabic (GA), maltodextrin (MD), and modified starch (MS) and their binary and ternary blends in order to encapsulate lemon oil via spray drying technique. They found that the percentage of MD in coating material impacts the properties of the encapsulated material. With the stability of 6 months, the spray-dried lemon oil showed better results in instant ice tea premix (Kausadikar et al., 2015).

Reineccius et al. (2002) studied the incorporation and retention of various spray-dried flavor compounds in □, □, and □-cyclodextrins (CyDs) during storage based on their volatility. Spray-dried capsules were stored at 20 or 40˚C and 65 or 80% relative humidity, whereas losses during the encapsulation process and the subsequent storage period were monitored analytically. Encapsulation using CyDs as wall material and via spray–drying showed an effective matrix entrapment as well as molecular inclusion.

Non-enzymatic browning and oxidation of limonene were investigated as two possible deterioration routes during encapsulated flavors› storage. Overall, the retention of flavors during drying with a limited loss of aldehydes and limonene during storage was improved with increase feed solids. The wall materials, GA, MS, sodium caseinate (SC) whey protein isolate, gave the highest flavor retention during drying; 94%, 88%, and 87%, respectively. However, only proteins effectively limited limonene oxidation, whereas >70% retained. Non-enzymatic browning was observed for all powders prepared with proteins, but no browning occurred with traditional materials like GA or MS(Charve &Reineccius, 2009).

Recently, Pratiwi et al. (2016) evaluated the microcapsule stability of cinnamon essential oil against storage temperature using MD and GA. The ratio of the essential oil to the wall material applied was 1:5. The formulated microcapsules were stored at 30-70 ˚C for 4 weeks. The highest microencapsulation efficiency of cinnamon essential oil was recorded in the sample coated with MD to GA with a ratio of 1:1.

In agreement with the above studies, from an economic point of view, foods and beverages containing encapsulated flavors may inevitably be stored for an extended period. Therefore, this study aimed to investigate the effect of storage on the overall stability, physical properties, and morphology of encapsulated aroma compounds belong to different chemical classes coated with ternary blends in different proportions.

## Materials and Methods

*Materials*

The present study was performed at the National Research Center from October 2016 to December 2017.The flavorings used during the study, isoamyl acetate, citral, allyl caproate, and linalool, were supplied from Sigma-Aldrich (St. Louis, MO), while Eng. Sherif Yahia (Life Essences, Cairo, Egypt) kindly provided the Orange oil. The coatingagents applied were GA (Avonchem, Cheshire, UK), MD with DE 12–15 (XINGMAO, Qingdao, Shandong, China), and SC (Fonterra New Zealand, Auckland).

*Methods*

*Preparation of emulsions*

The solutionsof GA (5–15% weight/volume w/v) and SC (5% w/v) werepreparedseparately by dissolving them in warm deionized water and left overnight at 4 °C to allow full hydration of the polymer molecules. Different MD concentrations (15–25.0% w/v) were then dissolved in the gum solutions followed by SC under constant stirring to end up with a total solid content of 35.0% w/v (Table 1). Tween80 (1.0% w/v, based on water) was dissolved in the coating solution, followed by the addition of flavorings to obtain a 15% w/v based on water feeding emulsion. After emulsification for 15 min with a magnetic stirrer (Bibby Scientific Ltd, U.K., 230V, 500W, 50 Hz), the whole mixture was homogenized for 20 min at full sonicator power (200.0 W, 24 kHz) using a UP200S ultrasound homogenizer (IKA Hielscher GmbH, Berlin, Germany). The mixture vessel was thermally isolated by iced water bath during the ultrasound homogenization to avoid any rise of emulsion temperature (Kausadikar et al., 2015 and Charve & Reineccius, 2009).

*Spray drying process*

The emulsions were spray-dried using co-current Mini Spray Dryer B-290 (Büchi, Flawil, Switzerland) equipped with a two-fluid nozzle atomizer of an internal tip with an opening of 0.7 mm in diameter and an external ring with an opening of 1.5 mm in diameter. The inlet and outlet temperatures were maintained at 160.0 °C and 80.0 °C (±1.0 °C), respectively.Moreover, the constant process parameters included a drying airflow rate of 85.0% of the suction fan controller. The drying process was conducted twice. The spray-dried samples were stored in desiccators at room temperature for one year.

**TABLE 1. Coating blends used to encapsulate flavorings.**

| Aroma Compound* | Wall materials (%) | | |
|---|---|---|---|
| | *Gum Arabic (GA)* | *Sodium Caseinate (SC)* | *Maltodextrin (MD)* |
| A1 - E1 | 5 | 5 | 25 |
| A2 - E2 | 10 | 5 | 20 |
| A3 - E3 | 15 | 5 | 15 |

*Where A: Linalool, B: Citral, C: Limonene, D: Allyl caproate, and E: Isoamyl acetate

*Characteristics of stored spray-dried flavors*

*Moisture content*

Analysis was conducted on 3–5 grams of the stored spray-dried flavorings at 105.0 °C until a constant weight was accomplished. The analysis was performed in triplicates, and the arithmetic means calculated ±SD (AOAC, 2005).

*Scanning Electron Microscopy (SEM)*

The morphology and the size of the stored microcapsules were evaluated using the field emission FE-SEM (Quanta FEG 250, FEI, Czech Republic). The stored samples were gold-sputtered by mounting on aluminum stubs with double-sided adhesive tape and coated with gold using an Edwards's sputter coater S150A (Crawley, England). FE-SEM images were taken with magnification ranges of 1000-15000x and an accelerating voltage of 10 kV.

*Non-enzymatic browning (NEB)*

The extent NEB or Maillard reaction in the fresh and stored,spray-dried flavorings was conducted by Minolta chromameter CR-200 (Minolta, Osaka, Japan)and was expressed as the *b** value. The total color change □*b** (□*b**= $b^*_{stored}$ - $b^*_{fresh}$) of the powders was used to reflect the extent of browning during storage. The total parameters measured during this analysis were: L* — expressed the lightness (in %), a* value — redness (positive (+ve)) to greenness (negative (-ve)), and b* value —yellowness (+ve) to blueness (-ve).

*Effect of storage on the chemical composition of the spray-dried flavorings during storage*

The evaluation of the chemical composition of the stored spray-dried flavorings was performed using gas chromatography-mass spectrometry (GC–MS). Approximately 0.5g of each stored spray-dried flavoring was dissolved in 2.8 g of distilled water and was mixed using a vortex mixer and then extracted with 4g of diethyl ether solution. The supernatant was stored in airtight glass vials covered with aluminum foil at 20°C until analysis (Charve&Reineccius, 2009).

*GC–MS analysis*

For GC, HP5890 (Agilent, CA, USA) was equipped withHP5970 mass spectrometer (Agilent, CA, USA) was used for analysis; injection temperature: 220°C;column: Agilent J&W DB-560m×0.32mm id×0.25µm film thickness (Agilent, CA, USA); oven temperature programming: 50 °C for 5 min, and then heating at 4 °C / min to 250 °C;carrier gas: helium at a flow rate of 1.1ml/min; split ratio 1:10; electron impact (EI) applied to obtain mass spectra; the ionization energy: 70eV; scan m/z range: 29 - 400amu; identifications were based on the comparison with respective retention indices based on a series of n-alkanes ($C_6$ -$C_{22}$), standards,and library of mass spectra (National Institute of Standard and Technology, MD, USA)(Adams, 2007).

*Statistical analysis*

The Statistical Package for the Social Sciences (SPSS 16) was applied, whereas the data were expressed as mean ± SD.

## Results and Discussion

Concerning the main objectives of this study mentioned earlier, additional considerations were taken into account to deal with the process's efficiency. Industrially, the highest dryer infeed solids are strongly recommended, not only for the maximum spray-dried yield but also for improving volatiles' retention throughout the drying process. Therefore, blends with total infeed solids of 50% were prepared during this study to simulate the real-world situation. In this study, emulsions are prepared with a viscosity of 171 cP as the maximum infeed viscosity that can be efficiently atomized in our spray dryer (Farouk, 2020). Such maximum infeed viscosity affected the design of ternary blends applied in this study, whereas 15% of the GA may replace SC and considered

the maximum allowed concentration. Charve &Reineccius (2009) reported that SC could be used due to its emulsifying properties with not more than 10% due to physical and economic reasons. Only 5% of SC could be used efficiently during the current study; therefore, GA was used to compensate for emulsifying properties of the blend prepared.

*The moisture content of the stored spray-dried flavors*

After storage microcapsules for 12 months at room temperature under desiccator, the water content of the microcapsules of spray-dried flavorings is ranged between 2.96 to 5.97% (Table 2). The obtained range of moisture content is lower than that proposed by Pratiwi et al. (2016) (5.11-6.69%) but following Reineccius(2004) (2-6%). In a previous study conducted by Farouk et al.(2020), a comparable range (2.9 – 5.15%) was recorded for the same spray-dried aroma compounds in a fresh state using the same wall materials (Table 2). During storage, the adsorbed water content of the microcapsules increased in a significant manner for both linalool and isoamyl acetate capsules. The hydrophilic and hydrophobic nature of flavorings is a crucial factor since the solubility of aroma compounds, e.g., linalool and isoamyl acetate, as well as the hydrogen bonds formed, may explain the higher moisture content upon storage (Soottitantawatet al., 2015). Meanwhile, the hygroscopic nature of GA does not significantly affect the water adsorbed percentage upon storage.

**TABLE 2 . Moisture content of aroma spray-dried powders before and after storage for one year (As is basis).**

| Aroma and wall mix No.* | Moisture % | |
|---|---|---|
| | Before storage | After storage |
| **A1** | $3.61^{M} \pm 0.05$ | $4.09^{K} \pm 0.08$ |
| **A2** | $5.15^{C} \pm 0.06$ | $5.97^{A} \pm 0.15$ |
| **A3** | $4.55^{G} \pm 0.10$ | $5.18^{C} \pm 0.12$ |
| **B1** | $3.62^{L} \pm 0.12$ | $3.74^{L} \pm 0.06$ |
| **B2** | $4.58^{M} \pm 0.01$ | $4.74^{F} \pm 0.04$ |
| **B3** | $3.74^{K} \pm 0.01$ | $3.80^{L} \pm 0.03$ |
| **C1** | $3.55^{HI} \pm 0.03$ | $3.60^{M} \pm 0.08$ |
| **C2** | $4.11^{N} \pm 0.03$ | $4.25^{IJ} \pm 0.03$ |
| **C3** | $4.32^{DE} \pm 0.01$ | $4.41^{H} \pm 0.05$ |
| **D1** | $3.13^{N} \pm 0.05$ | $3.22^{N} \pm 0.10$ |
| **D2** | $4.87^{DE} \pm 0.05$ | $4.97^{D} \pm 0.05$ |
| **D3** | $2.90^{O} \pm 0.03$ | $2.96^{O} \pm 0.04$ |
| **E1** | $4.16^{JK} \pm 0.06$ | $4.78^{EF} \pm 0.03$ |
| **E2** | $4.20^{JK} \pm 0.06$ | $4.90^{D} \pm 0.08$ |
| **E3** | $4.91^{D} \pm 0.06$ | $5.38^{B} \pm 0.12$ |
| **LSD 5%** | **0.11** | |

*A: Linalool, B: Citral, C: Orange oil, D: Allyl Caproate, and E: Isoamyl Acetate; 1,2, and 3 referred to wall mix number in Table 1.

*The stored powder morphology*

The surface of stored powder particles was investigated using SEM (Fig. 1A-E). More GA concentrationsand MD as the primary encapsulating agent kept the stored capsules inhomogeneous state with few wrinkles, larger agglomerates of irregular shapes, and smoother surface. The homogeneous nature of walls could also be due to the complete dissolving of SC and GA before the spray drying process. The formation of larger particles or agglomerates is attributed to the high viscosity of the feed emulsion, taking into consideration the direct relation between particle size and viscosity (Farouk et al., 2020). Some indentations were observed on the surface of particles obtained by spray drying. This could be referred to as the particle shrinkage upon the drastic loss of moisture followed by cooling (Saénz et al., 2009). The external particle surfaces of the stored microparticles were continuous, with little cracks, porosity, or breaks in contrast to the fresh ones reported before, which negatively affected the retention of the flavoring (Farouk et al., 2020). Characteristics like no cracks, porosity, or breaks are critical to ensure greater protection and retention of the active ingredient, as reported by Trindade & Grosso (2000), Bertolini et al.(2001) and Comunian et al. (2011). The SEM images before storing the spray-dried flavorings coated with the same wall blends reported in the current study showed that the predominant MD as a coating material provided homogenous, spherical, and smooth capsules. The high viscosity of the prepared emulsions containing GA caused some agglomerates with irregular shapes, which align with SEM images of the stored capsules (Soottitantawat et al., 2015).

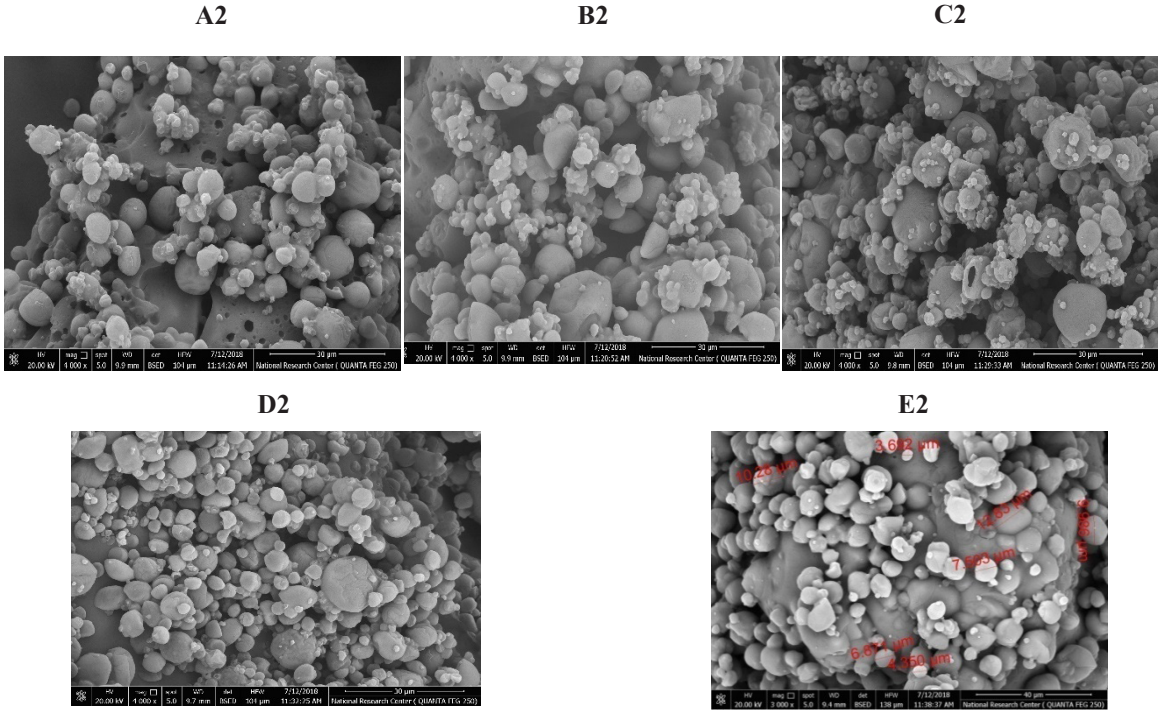


**Fig. 1. SEM of a stored spray-dried aroma (A: linalool, B: citral, C: Orange oil, D: allyl caproate, and E: isoamyl acetate; 1,2 and 3 referred to wall mix number in Table 1).**

*Non-enzymatic browning (NEB) of the stored spray-dried aroma:* According to the results of different CIE-LAB characteristics reported in Table 3, all spray-dried aroma except that containing limonene underwent significant changes in color (□*b**) during storage. According to Charve and Reineccius(2009), the presence of protein, e.g., SC, is responsible for the browning (□*b**)detected in the examined stored spray-dried samples, especially those containing carbonyl groups, e.g., citral or isoamyl acetate,while blends containing GA, modified starch or MD did not brown. Color changes related to the browning resulting from the interaction between aldehydes/ketones and the free amino groups of proteins (Schiff base formation-initiated NEB) with brown pigments

lead to the disappearance of aroma compounds. Earlier research reported different forms of binding between aldehydes, particularly 2-alkenals and proteins, accompanied by the browning of the reaction media. For example, partially reversible binding was observed with soy and whey proteins (Gremli, 1974; Zhou & Cadwallader, 2006), and recently, covalent binding of (*E*)-2-hexenal with lysyl and histidyl residues of whey protein and sodium caseinate (Meynier et al., 2004).

*Effect of storage on the chemical composition of the spray-dried flavorings during storage.*

The overall stability of the dried flavors during storage was assessed by interpreting the aroma chemical structure or composition using GC - MS. Thechanges in flavoringsstructure reflected the overall loss due to their reactivity with the environment during storage or through the effect of drying conditions. There is not any change in the chemical structure of the flavorings neither before nor after the storage during the present study. However, only a quantitative loss in the aroma constituents was observed due to their volatility and subsequent vapor pressures or interaction with the environment (Charve & Reineccius, 2009; Adams, 2007).

Figure 2illustrates the retention percentage of spray-dried flavorings after storage for one year. Three comparable loss profiles were identified among the compoundsstudied: citral, allyl caproate, and orange oil, respectively. By the end of the storage, isoamyl acetate recorded the highest retention,followed by linalool. Infeed solids significantly influenced volatileretention during storage,with a similar overall trend for all flavorings under investigation. Better retention overtime was achieved when GA was used as a significant wall constituent compared to MD at constant SC. This may have beendue to particle structure changes (e.g., less void volume orsurface cracks) when spray-dried at higher feed solids(Charve & Reineccius, 2009).

**TABLE 3. Colour parameters of stored spray-dried powders before and after storage for one year.**

| Aroma and wall mix No.* | Before storage | | | After storage | | | |
|---|---|---|---|---|---|---|---|
| | L* | a* | b* | L* | a* | b* | |
| **White blank** | 50.68 | -9.26 | 9.21 | | | | |
| **Black blank** | 50.86 | 1.60 | 25.94 | | | | $\Delta b^*$ |
| **A1** | $96.89^{A}\pm0.30$ | $-0.46^{LMN}\pm0.01$ | $6.20^{T}\pm0.45$ | $95.33^{DE}\pm0.39$ | $-0.48^{LMN}\pm0.01$ | $7.99^{OP}\pm0.16$ | 1.79 |
| **A2** | $95.6^{CD}\pm0.31$ | $-0.34^{H}\pm0.01$ | $7.74^{PQ}\pm0.38$ | $94.15^{G}\pm0.01$ | $-0.36^{HIJ}\pm0.01$ | $9.73^{K}\pm0.01$ | 1.99 |
| **A3** | $94.96^{F}\pm0.06$ | $-0.38^{HIJK}\pm0.02$ | $8.92^{N}\pm0.10$ | $93.95^{G}\pm0.09$ | $-0.42^{IJKL}\pm0.04$ | $9.88^{K}\pm0.07$ | 0.97 |
| **B1** | $91.28^{L}\pm0.12$ | $0.67^{D}\pm0.07$ | $22.67^{B}\pm0.03$ | $90.06^{N}\pm0.09$ | $0.74^{D}\pm0.11$ | $25.03^{A}\pm0.15$ | 2.36 |
| **B2** | $89.86^{NO}\pm0.42$ | $2.46^{B}\pm0.13$ | $18.71^{D}\pm0.55$ | $88.37^{P}\pm0.02$ | $2.89^{A}\pm0.03$ | $22.25^{C}\pm0.02$ | 3.54 |
| **B3** | $90.09^{N}\pm0.24$ | $2.39^{C}\pm0.11$ | $14.47^{F}\pm0.41$ | $89.60^{O}\pm\pm0.18$ | $2.46^{BC}\pm0.09$ | $18.65^{D}\pm0.26$ | 4.18 |
| **C1** | $95.71^{C}\pm0.06$ | $-1.19^{P}\pm0.02$ | $10.83^{J}\pm0.09$ | $96.7^{A}\pm0.13$ | $-0.53^{NO}\pm0.05$ | $7.82^{Qv}0.19$ | -3.01 |
| **C2** | $94.87^{F}\pm0.25$ | $-1.20^{P}\pm0.01$ | $13.55^{G}\pm0.37$ | $95.09^{EF}\pm0.02$ | $-0.35^{HI}\pm0.01$ | $9.57^{KL}\pm0.02$ | -3.97 |
| **C3** | $90.90^{M}\pm0.26$ | $-0.46^{LM}\pm0.03$ | $15.19^{E}\pm0.22$ | $91.71^{K}\pm0.22$ | $-0.24^{F}\pm0.07$ | $12.91^{H}\pm0.18$ | -2.28 |
| **D1** | $96.65^{A}\pm0.06$ | $-0.59^{O}\pm0.01$ | $6.29^{T}\pm0.10$ | $95.81^{BC}\pm0.14$ | $-0.51^{MN}\pm0.04$ | $7.327^{RS}\pm0.15$ | 1.03 |
| **D2** | $95.55^{CD}\pm0.01$ | $-0.44^{KL}\pm0.01$ | $7.61^{QR}\pm0.10$ | $94.96^{F}\pm0.05$ | $-0.32^{GH}\pm0.02$ | $9.11^{MN}\pm0.01$ | 1.55 |
| **D3** | $95.60^{CD}\pm0.17$ | $-0.38^{HIJK}\pm0.02$ | $7.74^{PQ}\pm0.02$ | $94.80^{F}\pm0.18$ | $-0.26^{FG}\pm0.04$ | $8.30^{O}\pm0.33$ | 0.44 |
| **E1** | $95.57^{CD}\pm0.11$ | $-0.43^{JK\ L}\pm0.02$ | $4.60^{V}\pm0.30$ | $93.66^{H}\pm0.12$ | $-0.23^{F}\pm0.07$ | $9.32^{LM}\pm0.18$ | 4.56 |
| **E2** | $96.07^{B}\pm0.05$ | $-0.26^{FG}\pm0.01$ | $5.64^{U}\pm0.28$ | $92.75^{I}\pm0.01$ | $-0.03^{E}\pm0.02$ | $11.22^{I}\pm0.01$ | 5.43 |
| **E3** | $94.19^{G}\pm0.07$ | $-0.45^{KLM}\pm0.01$ | $7.21^{S}\pm0.05$ | $95.33^{DE}\pm0.09$ | $-0.48^{LMN}\pm0.04$ | $7.99^{OP}\pm0.15$ | 2.52 |
| **LSD 5%** | **0.29** | **0.07** | **0.34** | **0.29** | **0.07** | **0.34** | |

*A: Linalool, B: Citral, C: Orange oil, D: Allyl Caproate, and E: Isoamyl Acetate; 1,2 and 3 referred to wall mix number in Table 1.

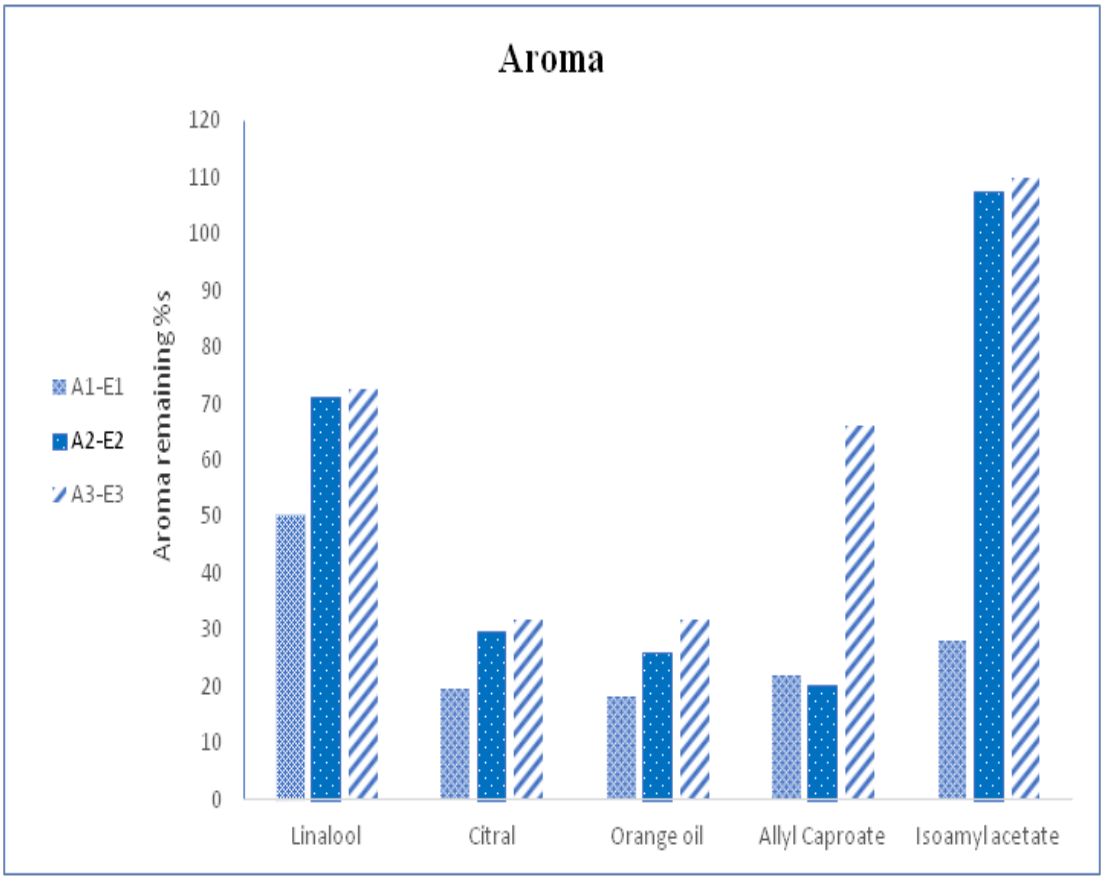


**Fig. 2. Individual retention of spray-dried aroma compounds during storage (*A: Linalool, B: Citral, C: Orange oil, D: Allyl Caproate and E: Isoamyl Acetate; 1, 2, and 3 referred to wall mix number in Table 1).**

## Conclusions

Nature of flavoring as well as wall material affected the retention of the spray-dried aroma during storage. By the end of the storage, isoamyl acetate recorded the highest retention, followed by linalool, while citral and Orange oil were the lowest. However, the morphology of the resulted microcapsules and their moisture content were influenced by the wall combination. The browning in the encapsulated stored samples containing isoamyl acetate recorded the highest intensity compared to the others on storage, followed by Orange oil, linalool, allyl caproate, and citral, respectively. Concerning their emulsification properties, SC showed a negative effect on the browning of the spray-dried flavorings, while GA: MD (1:1w:w) showed good retention, efficiency, and morphology of the flavorings and capsules. Chemical and physical analysis of the spray-dried microcapsules after storage revealed the possibility of using spray-dried flavors in the food matrix for a long time without affecting the chemical structure or the aroma of such flavorings.

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