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Spray Drying Encapsulation of Thyme Essential Oil for Potential Applications in the Meat Industry

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ABSTRACT

In the present study, encapsulation of thyme essential oil (TEO) through spray drying was done using two different wall materials viz Arabic gum (AG) and maltodextrin (MD) at two different concentrations viz. 12 and 14%, respectively, with 3% carrageenan as reinforcing material. The encapsulated powders obtained were designated AGc12, AGc14, MDc12 and MDc14. The encapsulation was carried out by spray drying at three inlet temperatures (120˚C, 140˚C, and 160˚C) and various properties of spray-dried encapsulated powder were studied, viz. encapsulation efficiency (EE), residual moisture content, bulk and tapped density. With an increase in inlet temperature, a significant ($P<0.05$) increase in encapsulation efficiency was observed, and it also varied with the type of wall material used. The encapsulation efficiency at different inlet temperatures and with use of different wall materials at two concentrations varied from 12.23% to 78.66%. The type of wall material had a significant ($P<0.05$) effect on residual moisture content and was observed to be highest for powders with AG as wall material than MD. Both bulk and tapped density were also dependent on wall material concentration as well as type of wall materials used. Based on encapsulation efficiency, bulk density and tapped density, AGc14 as the wall material, i.e., Arabic gum at 14% concentration and a 160 °C inlet temperature, was selected as the spray-drying conditions and parameters for encapsulation of TEO.

HIGHLIGHTS

- Spray drying conditions significantly influenced the encapsulation efficiency and other properties of thyme essential oil microcapsules.
- Gum arabic (14%) wall matrix dried at high temperature produced microcapsules with superior encapsulation efficiency.

Keywords: Encapsulation, gum Arabic, maltodextrin, Thyme essential oil, carrageenan, spray drying

Rising consumer interest in natural food preservatives has boosted research into plant-based essential oils, valued for their antimicrobial, antioxidant, antifungal and flavour-enhancing qualities (Bhat *et al*., 2025). Thyme (*Thymus vulgaris* L.) essential oil stands out as a highly effective natural antimicrobial, largely due to its high levels of thymol and carvacrol, which provide broad-spectrum activity against foodborne pathogens and spoilage microbes (Punya *et al*., 2019). However, incorporating thyme oil directly into food remains difficult because of its high volatility, vulnerability to oxidation, poor water solubility and sensitivity to heat, light and oxygen, which cause rapid loss of activity during processing and storage (Altay *et al*., 2024). These limitations hinder its industrial use, underscoring the need for stabilization methods. Encapsulation is known to be as a promising technique to enhance the stability, handling and performance of essential oils in food systems. Among the available encapsulation techniques, spray drying is the most widely adopted in

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the food industry because it is economical, continuous, scalable and capable of producing free-flowing powders with high retention of volatile compounds. The technique offers improved oxidative stability, protection against environmental degradation, controlled release of bioactive compounds and enhanced shelf life while allowing easy incorporation of essential oils into food matrices (Altay *et al*., 2024; Phanse and Chandra, 2024). However, optimization of spray-drying process variables is essential for maximizing encapsulation efficiency and improving powder quality. The encapsulation performance of spray-dried powders is largely governed by the physicochemical properties of the wall materials. Gum arabic is widely regarded as an excellent encapsulating agent because of its superior emulsifying capacity, low viscosity and excellent film-forming characteristics, whereas maltodextrin contributes desirable drying behaviour, improved solubility, low hygroscopicity and reduced production cost (Alves *et al*., 2014). Alongside, the concentration of wall materials and spray drying temperatures also play a critical role in affecting the quality of spray-dried powders. These parameters influence droplet drying kinetics, moisture removal, particle morphology, bulk and tapped density, encapsulation efficiency and retention of volatile compounds. Therefore, careful optimization of spray-drying conditions is essential to obtain stable microcapsules with desirable physicochemical characteristics (Kori *et al*., 2022). While several studies have explored spray drying of essential oils, there is limited systematic data on how wall material concentration and inlet drying temperature together affect the encapsulation properties of thyme essential oil. This study examined how variations in wall-material concentrations and inlet air temperatures affect key factors, including encapsulation efficiency, residual moisture, bulk density and tapped density of spray-dried thyme oil powders. Additionally, it aimed to determine optimal processing conditions for creating thyme essential oil microcapsules with enhanced physicochemical qualities appropriate for food use.

MATERIALS AND METHODS

Thyme essential oil: Source and composition

Thyme essential oil (TEO) was obtained from Moksha Lifestyles Products, New Delhi, India. It appeared clear, ranging from yellow to dark red. The main active components were γ-terpene (>30%), Thymol (29.10%) and p-cymene (>20%). Its specific gravity was 0.88, with a refractive index between 1.49 and 1.51 at 20°C.

Wall materials and spray drying operating conditions

The feed for the Buchi Spray Dryer B-290 was prepared by dissolving wall materials and surfactant in distilled water. Arabic gum (AG) and maltodextrin (MD) were dispersed in hot distilled water (50-55°C) with magnetic stirring until fully dissolved, then left for one hour to hydrate. Then, Tween-80 as a surfactant was added at 1%. The TEO was added dropwise in the wall material solution under continuous stirring and homogenised at 5500 rpm using Ultra Turrax homogeniser. The obtained solutions were atomised using laboratory spray dryer (Buchi Spray Dryer B-290). Arabic Gum (AG) and Maltodextrin (MD) served as wall materials at two levels i.e. 12% and 14%, across three inlet temperatures (120°C, 140°C and 160 °C) for spray-drying encapsulation of Thyme essential oil. Additionally, Carrageenan was used as a reinforcing wall material at 3% with both of them. The aspirator flow rate was kept 100% and the feed pump speed was 6-10 ml/min. The encapsulated powders so obtained were tagged as AGc12, AGc14, MDc12 and MDc14 (Table 1) and kept at 4 °C till analyzed.

Table 1: Different treatments for the encapsulation of TEO through spray drying

Treatments	Type of wall material	Concentration
AGc12	Arabic Gum + carrageenan	12%
AGc14	Arabic Gum + carrageenan	14%
MDc12	Maltodextrin + carrageenan	12%
MDc14	Maltodextrin + carrageenan	14%

Analytical parameters

Encapsulation efficiency

Surface oil content was measured following the method by Ton *et al.* (2016). In brief, 1 g of spray-dried powder was accurately placed into an extraction flask to which 25 ml of petroleum ether (boiling point 40–60°C) was added. The mixture was vigorously shaken for 10 minutes, then filtered through a cloth. The filtrate was collected in a pre-weighed petri dish and dried at 102 ± 2°C for 1 hour. After cooling in a desiccator to room temperature, the dish was weighed and the surface oil content was calculated from the weight increase. Total oil content was determined using the same method by Ton *et al*. (2016). The amount of encapsulated oil was obtained by subtracting surface oil from total oil. Encapsulation efficiency was then calculated with the following formula:

$$\textit{Encapsulation efficiency (\%)} = \frac{\textit{Total oil content} - \textit{Surface oil content}}{\textit{Total oil content}} \times 100$$

Residual moisture content

It was calculated using the method given by Lakshanasomya *et al.* (2011) by drying at 110±2°C in hot air oven until constant weight.

Bulk and tapped density

It was calculated by using the method of Jinapong *et al.* (2008). The powders were carefully loaded into a 100 ml tared graduated cylinder filled to the 100-ml mark and weighed. The volume directly read from the cylinder was then used to determine the bulk density (d) using the mass/volume formula. Tapped density was measured following the method outlined by de Barros Fernandes *et al.* (2014). About 5 g of powder was poured into a 25-ml graduated cylinder, which was repeatedly tapped by lifting and dropping under its own weight until the volume readings stabilized with negligible differences. Using the mass (m) and the apparent (tapped) volume (V), the bulk density was calculated as m/V (g/cm^3).

Statistical analysis

Data analysis was conducted with SPSS-24.0 (SPSS Inc., Chicago, II USA). Each experiment was performed three times with samples collected in duplicates (*n*=6) per parameter. Significance was assessed at the 5% level (P<0.05) using Duncan's Multiple Range Test (DMRT).

RESULTS AND DISCUSSION

Encapsulation efficiency (EE) (%) of spray-dried encapsulated thyme essential oil (eTEO) powder by using Arabic gum and Maltodextrin as wall material

The results for encapsulation efficiency of eTEO obtained using two different wall materials viz. Arabic Gum (AG) and Maltodextrin (MD) at 12% and 14% concentrations i.e., AGc12, AGc14, MDc12 and MDc14, spray-dried at three different inlet temperatures (120˚C, 140˚C, and 160˚C) are presented in Fig. 1.

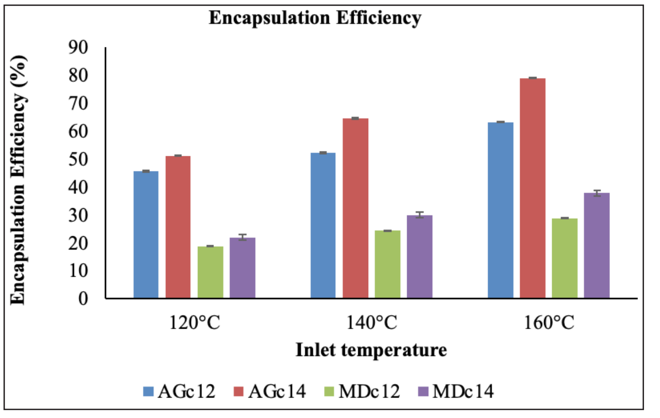

Fig. 1: Effect of different polymers, their concentration and inlet temperature on encapsulation efficiency (%) of encapsulated thyme essential oil (eTEO) (Mean ± S.E.); *n* = 6

In essential oil encapsulation, encapsulation efficiency (EE) is a key quality measure, representing the percentage of active component trapped relative to the total initial essential oil amount. Several factors affect EE and successful encapsulation results in a higher encapsulation efficiency percentage. Of the two wall materials tested, AGc demonstrated higher (P<0.05) encapsulation efficiency than MDc, irrespective of wall material concentration. Similar findings have also been reported by Akram *et al*. (2021) who found that Gum arabic-based microcapsules exhibited higher encapsulation efficiency along with better storage stability for fish and flexseed oils as compared to maltodextrin and their combinations. Silva *et al.* (2014) studied themicroencapsulation of green coffee oil by spray drying using different combinations of wall materials viz. modified starches (Hi-Cap, Capsul and N-Lok) or gum arabic with maltodextrin (MD) (75:25) and found that encapsulation efficiency was highest amongst microcapsules with Arabic gum as wall material. Premi and Sharma (2017) also demonstrated that Arabic gum improved the encapsulation behavior of spray dried drumstick oil as compared to whey protein concentrate and maltodextrin. In general, out of all the wall materials employed for encapsulation, Arabic gum is most popular choice owing to higher emulsifying capacity, leading for formation of stable emulsions and better spray dried products (Jafari *et al*., 2008). As far as maltodextrin as a wall material is concerned, it has lower emulsifying capacity than Arabic gum. Addition of carrageenan in both the polymeric solutions helped in increasing clear viscosity of solution and hence helped in increasing the encapsulation efficiency. It would have led to formation of a dry crust around the microcapsules during drying and ultimately hindered the core material to escape leading to higher encapsulation efficiency.

The impact of the concentration of wall materials on encapsulation efficiency is depicted in Fig. 1. An increase in wall material concentration led to a significant (P<0.05) increase in encapsulation efficiency, irrespective of type of wall material. This could be attributed to increase in total solid concentration of matrix to be encapsulated. It may have further enhanced oil retention during spray drying by shortening the time needed to form a semi-permeable membrane on the particle's surface. Similar findings have been reported by Ton *et al.* (2016) who reported that encapsulation efficiency was positively influenced by total wall material concentration. Chew *et al.* (2018) also reported that with increase in solid content there was a consequent increase in encapsulation efficiency.

The encapsulation efficiency at different inlet temperatures and with use of different wall materials at two concentrations varied from 12.23% to 78.66%. With an increase in inlet temperature, a significant (P<0.05) increase in EE was observed, irrespective of type of biopolymer and concentration of the same. It could be due to faster rate of evaporation at higher temperatures, leading to formation of semipermeable membrane around it which might occlude the movement of core material from the microcapsules.

Moisture content (%) of spray dried encapsulated thyme essential oil powder by using Arabic gum and Maltodextrin as wall material at three different inlet temperatures

A powder's residual moisture level influences its shelf life, stability, solubility, flow characteristics and overall acceptability (Sanchez-Reinoso *et al.,* 2017). It is a key factor indicating the success of spray drying. Excess moisture reduces functional performance and promotes quicker oxidation and microbial growth. According to BIS standards, the total moisture content of spray-dried powder must be below 4%.

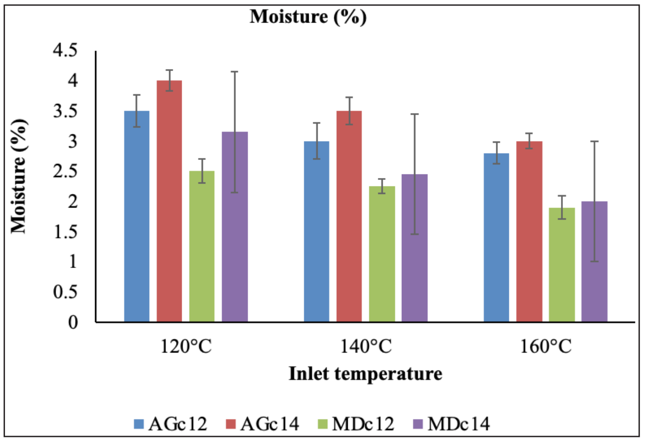

Fig. 2: Effect of different polymers, their concentration and inlet temperature on moisture content (%) of encapsulated thyme essential oil. (Mean ± S.E.); n = 6

Fig. 2 illustrates the impact of two wall materials, AG and MD, on the residual moisture content of spray-dried encapsulated powder. The type of wall material significantly influenced moisture levels (P<0.0.5), with powders containing AG showing higher residual moisture than those with MD. This difference may be attributed to the lower emulsion viscosity of maltodextrin. Arabic gum has a ramified structure that enhances thickening and increases viscosity, leading to rapid drying and the formation of a crust around the particles. This crust restricts water movement, resulting in higher residual moisture. Additionally, Arabic gum's hygroscopic nature causes it to absorb moisture from the environment, further increasing residual moisture content (Carneiro *et al.*, 2013). Residual moisture content increased with higher wall material concentrations, regardless of the material type (Fig. 2). This could be because larger molecular sizes of the carrier agent slowed water diffusion during spray drying. Additionally, higher wall material levels may have accelerated shell formation around encapsulated particles, potentially blocking water molecule migration. Similar results were reported by Karrar *et al*. (2021). The inlet temperature during spray drying significantly (P<0.05) influences the residual moisture content of the powder. Increasing the inlet air temperature from 120°C to 160°C leads to a significant reduction (*P*<0.05) in residual moisture, regardless of wall material type or concentration. This effect likely results from a faster evaporation rate from the emulsion, reducing moisture content in the powder. Additionally, the temperature difference between the particles and the drying medium may also contribute to the trend.

Density (gm/cm^3) of spray-dried encapsulated thyme essential oil powder (e-TEO) by using Arabic gum and Maltodextrin as wall material at three different inlet temperatures

Density is a crucial factor influencing the quality of encapsulated spray-dried powders. Typically, two types of density are examined in powders. Bulk density refers to the ratio of the mass of an untapped powder sample to its volume, including interparticulate voids. A higher bulk density suggests greater stability during storage, likely due to less air in the powder. This density is affected by the residual moisture content of the powders. In this study, AG exhibited a higher bulk density than MD among the two wall materials, primarily due to its noticeably greater moisture retention. This aligns with Raigarand Mishra (2015), who observed an increase in bulk density as moisture content rises, since higher moisture causes particle clumping, thereby raising bulk density. An increase in wall material concentration significantly (*P*<0.05) boosted bulk density, likely due to the higher total solid content. Similar results have been reported by Chew *et al*. (2018) and Sanchez-Reinoso *et al.* (2017). Additionally, bulk density depends on the inlet temperature during spray drying; higher temperatures tend to produce powders with lower bulk density, probably because of reduced residual moisture. These findings concur with Sanchez-Reinoso *et al.* (2017). Along similar lines, tapped density, which influences powder properties, showed a trend akin to bulk density. Powders with Arabic gum as the wall material had higher tapped density owing to increased residual moisture. Elevating the wall material concentration from 12% to 14% significantly (*P*<0.05) increased tapped density, likely due to moisture differences. Conversely, higher inlet temperatures during spray drying resulted in powders with lower tapped density, due to increased moisture evaporation, leading to reduced residual moisture; consistent with findings of Goula and Adamopoulas (2010).

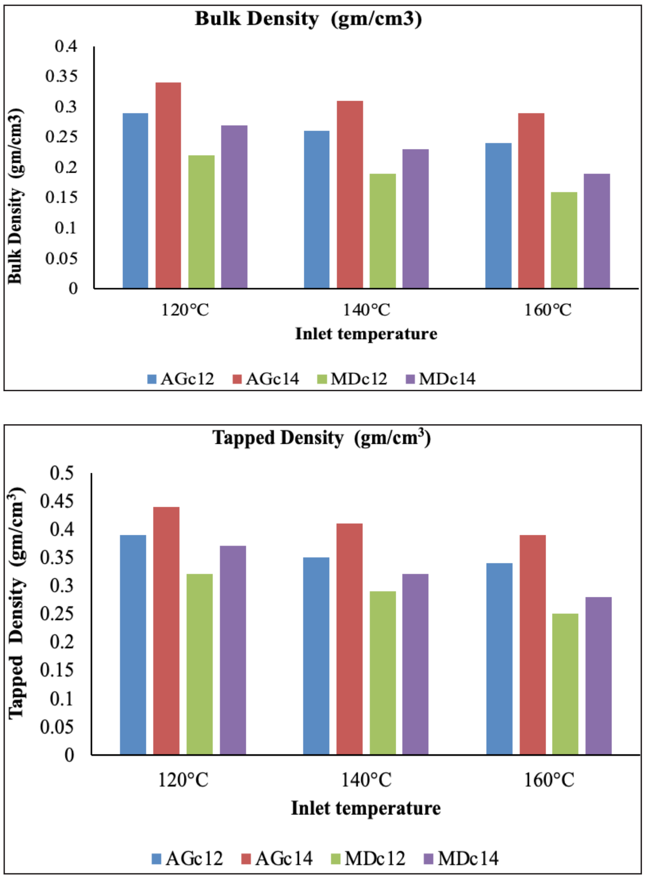

Fig. 3: Effect of different polymers, their concentration and inlet temperature on Bulk and tapped density of encapsulated thyme essential oil. (Mean ± S.E.); *n* = 6

CONCLUSION

The study explored how variations in polymers, their concentrations and inlet temperatures during spray drying influence encapsulation efficiency, residual moisture and bulk and tapped densities. Findings indicated that higher concentrations of Arabic gum produce powders with superior encapsulation efficiency compared to maltodextrin. Furthermore, Arabic gum powders exhibit greater densities than other materials. It can be concluded that employing 14% Arabic gum at an inlet temperature of 160°C is effective for encapsulating thyme essential oil through spray drying, with promising applications in food systems for antimicrobial and antioxidant action.

GENERATIVE AI DECLARATION

The author(s) confirm that there was no use of artificial intelligence (AI)-assisted technology for assisting in the writing or editing of the manuscript, and no images were manipulated using AI. The authors developed and verified all scientific content, data analysis, interpretation of results, and conclusions. The authors take full responsibility for the accuracy, integrity, and originality of the work presented, and no AI tool was listed as an author.

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