

# Spray-dried maltodextrin- $\gamma$ -cyclodextrin microcapsules of garlic essential oil: Release kinetics and application in shelf-life extension of *Nem Chua Hue*

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## ABSTRACT

In this research, garlic essential oil (GEO) was microencapsulated by spray drying, and the use of the encapsulated powder in *Nem chua Hue* (a traditional fermented pork product) was studied. The experiments were carried out with three parameters: carrier types ( $\alpha$ -cyclodextrin,  $\gamma$ -cyclodextrin, maltodextrin- $\alpha$ -cyclodextrin, and a maltodextrin- $\gamma$ -cyclodextrin [MD- $\gamma$ -CD]), added GEO 2–8% (w/w), and spray drying temperature 160–180°C. The formulation having 5% (w/w) GEO and a MD- $\gamma$ -CD carrier with spray-dried temperature of 170°C gave the highest encapsulation efficiency and encapsulation yield of 91.86% and 80.53%, respectively. Fourier-transform infrared, X-ray diffraction, thermogravimetric analysis, and scanning electron microscopy analyzes confirmed the formation of an inclusion complex and showed a good structural stability of the microcapsules. The release behavior at humidity levels of 6–98% RH and temperature from 30 to 100°C fitted closely with the Avrami model ( $n \approx 0.30$ – $0.44$ ). When adding 0.01–0.02% (w/w) microencapsulated GEO into *Nem chua Hue*, the total aerobic count and total yeast and mold were significantly reduced, while lactic acid bacteria maintained at an appropriate level to support the fermentation process. There was also slowed acidification and increased product firmness. The 0.015% level could provide the best balance between sensory quality and microbiological safety. Its shelf-life was extended by at least 2 days. The MD- $\gamma$ -CD is effective for controlled release and potential for meat product preservation.

## 1. INTRODUCTION

Garlic essential oil (GEO) is a natural source that is rich in volatile sulfur compounds. It is famous for strong biological activities such as antibacterial, anti-inflammatory, antioxidant, and antifungal capacity [1]. Due to these characteristics, GEO is a potential natural food additive for improving flavor and preservation. However, sulfur compounds of GEO are unstable and easily lost during processing and storage. Therefore, this instability prevents its direct use in industrial food applications [2].

To solve these problems, microencapsulation is an effective strategy to protect sensitive bioactive compounds. It can minimize flavor loss and control the release of the active compounds [2]. Among

various microencapsulation methods, spray drying is widely used in the food industry because it is simple, cheap, and suitable for large-scale production. In addition, understanding how GEO is released from microcapsules is important in real food making. Environmental factors such as temperature and relative humidity (RH) can strongly influence the release process. The Avrami kinetic method is commonly used to describe release behavior and to explain the related release mechanisms. By linking Avrami parameters with storage conditions, it is possible to predict product stability and performance in food systems [3,4].

*Nem chua Hue* is a traditional Hue fermented pork product and famous for its distinctive aroma and taste. Like other kinds of *Nem chua* in other places of Vietnam, *Nem chua Hue* has a quite short shelf-life. Therefore, adding GEO microcapsules into *Nem chua Hue* could give some benefits, such as extending its shelf-life via antimicrobial activity and alleviating the pungent odor intensity that comes from fresh garlic – the topping in *Nem chua Hue*. This approach could enhance both food safety and consumer acceptability. Although microencapsulation of essential oils has been studied in many previous researches,

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application of GEO microcapsules in a traditional fermented product like *Nem chua Hue* is still limited, especially in the Vietnamese context.

Carrier materials or wall materials play a key role in encapsulation by protecting the core, controlling the release, and applying in food systems. Previous studies have shown that combinations of maltodextrin (MD) and cyclodextrins (CDs) are good for improving encapsulation efficiency and functional properties. For example, MD-CD systems have been used to reduce bitterness in whey protein hydrolysates [5], protect phenolic compounds [4,6], and keep color stability in fruit juices [7]. Therefore, using a dual-carrier system is expected to improve both encapsulation efficiency and functional performance of GEO. Accordingly, the present study was designed with the following specific objectives:

- (i) To optimize the encapsulation process of GEO by spray drying
- (ii) To evaluate the retention of the bioactivity of microencapsulated GEO (MGEO)
- (iii) To model the release kinetics using the Avrami equation and establish the relationship between kinetic parameters and RH/temperature conditions
- (iv) To apply the obtained powder to *Nem chua Hue* to assess its effectiveness in extending its shelf-life and sensory acceptability.

By addressing both fundamental and applied aspects, this study contributes to the development of safe, high-quality, and economically valuable food products. Furthermore, it provides a robust methodological framework for integrating natural bioactive compounds into functional foods, aligning with global efforts toward sustainable food preservation and reducing reliance on synthetic additives.

## 2. MATERIALS AND METHODS

### 2.1. Materials

Garlic was collected from Chieng Dong commune, Yen Chau district, Son La province (Vietnam). Garlic was finely ground and mixed with water at a ratio of 1:3. The mixture was sonicated for 10 min and then subjected to hydrodistillation for 3 h. The GEO was contained in a dark-colored bottle and stored in a fridge before doing encapsulation experiments.

The ingredients for *Nem chua Hue* preparation, including pork meat, pork skin, spices, and banana leaves, were procured from local suppliers. MD (DE = 10–12, moisture content 3–5%),  $\alpha$ -CD, and  $\gamma$ -CD (Wacker Chemical Corp., USA; moisture content 3–5%) were employed as wall materials. Analytical grade reagents, potassium sulfate ( $K_2SO_4$ ), sodium chloride (NaCl), potassium carbonate ( $K_2CO_3$ ), and silica gel, were also used.

### 2.2. Equipment

Some main equipment used for this research are presented in Table 1S.

### 2.3. Analysis Methods

#### 2.3.1. Chemical Composition Analysis of GEO

GEO was collected by hydrodistillation with the conditions mentioned in 2.1. Gas chromatography-mass spectrometry (GC-MS) analysis of the chemical composition was performed using a DB-1 capillary column (30 m  $\times$  250  $\mu$ m  $\times$  0.25  $\mu$ m). The injection port was maintained at 250°C, with a 1  $\mu$ L injection volume, helium as the carrier gas at a flow rate of 1 mL/min. The oven temperature was programmed as follows: An initial temperature of 45°C (held for 0 min), followed

by an increase at 5°C/min–150°C (held for 0 min), and then a ramp of 40°C/min–290°C (held for 5 min). Mass spectrometric detection was conducted in scan mode over an m/z range of 30–500, with the MS source and quadrupole temperatures set at 230°C and 150°C, respectively.

#### 2.3.2. Microencapsulation of GEO by Spray Drying

GEO was blended with the solution of the carrier materials ( $\alpha$ -CD,  $\gamma$ -CD, a mixture of MD and  $\alpha$ -CD, or a mixture of MD and  $\gamma$ -CD) at specified ratios (2%, 5%, and 8% GEO) and distilled water. The mixture was homogenized before spray-drying at different inlet temperatures (150–180°C) with a rotary atomizer.

#### 2.3.3. Characteristics of MGEO Powder

The characteristics of MGEO powder were evaluated through several analytical parameters. Moisture content was determined using the gravimetric method, while solubility was assessed following the procedure of Eastman and Moore [8]. Encapsulation yield was defined as the ratio of encapsulated essential oil amount in microcapsule and initial essential oil amount. Encapsulation efficiency was calculated from the proportion of essential oil retained within the microcapsules relative to the added initial amount [9], with the essential oil content quantified by measuring absorbance at 308 nm using a ultraviolet-visible spectrophotometer. Structural properties of the microcapsules were further examined using fourier-transform infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA), X-ray diffraction (XRD), microscopy, and scanning electron microscopy (SEM).

#### 2.3.4. Release Kinetic Studies

##### 2.3.4.1. Experimental Setup

The MGEO powders (0.1 g) were put into mighty vials covered by perforated aluminum foil and then were stored in sealed plastic chambers (29.5 cm  $\times$  19.5 cm  $\times$  20 cm) at different RH and temperature (T) conditions [10]. The RH levels of 6%, 40%, 75%, and 98% were maintained using silica gel, saturated salt  $K_2CO_3$ , saturated salt NaCl, and saturated salt  $K_2SO_4$ , respectively, for a period of 13 days [11,12]. The MGEO powders were taken out of the plastic box at specific intervals and estimated the remaining GEO content.

Similarly, to investigate the effect of temperature, the MGEO powders were stored under ambient RH conditions and subjected to three temperature levels: 30°C, 65°C, and 100°C. The storage experiment was conducted over a period of 35 days.

##### 2.3.4.2. Modeling

The release data were fitted using the Avrami model to calculate the kinetic constants k and n. This model was used to explain the release mechanisms and their relationship with RH and temperature [3].

#### 2.3.5. Application to *Nem chua Hue*

The experimental design includes preparing *Nem chua Hue* samples that were supplemented with MGEO at concentrations of 0.01%, 0.015%, and 0.02%. Meanwhile, the controlled samples contained a slice of fresh garlic (like the original method).

Physicochemical properties, including pH and firmness, were measured during the fermentation and storage periods. Microbiological assessments were done by counting total aerobic bacteria, lactic acid bacteria (LAB), and yeasts and molds. Sensory evaluation was conducted using a 9-point Hedonic scale (1 = dislike extremely, 9 = like extremely) to score color, aroma, texture, taste, and overall acceptability. The sensory panel consisted of 40 individuals (aged 18–23) who were familiar with *Nem chua Hue*. The panelists were

briefly trained before evaluation to ensure consistency in scoring. Samples were labeled with three-digit random codes and presented in a randomized order to minimize bias. Evaluation was performed in individual booths, and water was provided between samples as a palate cleanser [13].

**2.4. Statistical Analysis**

The experiments were all carried out in triplicate, and the data were analyzed using OriginPro 2024 and IBM the Statistical Package for the Social Sciences Statistics 20. Analysis of variance, followed by Duncan’s multiple range test, was used to determine statistically significant differences among samples. Release kinetic parameters were obtained by fitting the experimental data to Avrami model.

**3. RESULTS AND DISCUSSION**

**3.1. Components of GEO**

GC-MS analysis showed that the GEO contained organosulfur compounds that are typically found in GEO. The major constituents included di-2-propenyl trisulfide (DATS, 20.42%), diallyl disulfide (DADS, 11.07%), and methyl 2-propenyl trisulfide (11.24%), along with other dithiine and tetrasulfide derivatives, as can be seen in Table 1. These findings mean that trisulfide and disulfide compounds are the characteristic and predominant components of the GEO in this study.

Based on the composition, DATS (20.42%) and DADS (11.07%) were the 2 main compounds. These organosulfur substances are famous for their strong biological activities, such as antioxidant, anticancer, and antimicrobial effects. The high content of trisulfide and tetrasulfide derivatives (including DATS, methyl-2-propenyl trisulfide, di-2-propenyl tetrasulfide, and allyl propyl trisulfide) means that the GEO is rich with its multi-sulfur compounds, and these compounds are generally more biologically active than disulfides.

In addition, the presence of 2-vinyl-4H-1,3-dithiine and 3-vinyl-1,2-dithiacyclohex-4-ene is also remarkable for contributing both to the characteristic aroma and to the pharmacological properties of garlic. Minor constituents (e.g., allyl methyl disulfide and 1,2-dithiolane), although present at low levels but they may still play an important role due to synergistic effects. Dehariya *et al.* reported that GEO extracted using the Soxhlet system with ethanol contained the same major compounds as those found in this study, although the proportions were different. In their work, DADS, DAS, and allyl methyl trisulfide were the major components, while DATS accounted for only 3.46% [14]. In the same way, Satyal *et al.* also identified DADS, DATS, and allyl methyl trisulfide as the main compounds in GEO. Differences in composition and relative content may come from differences in extraction methods and origin of the raw material [15].

**3.2. Effect of Encapsulation Parameters on the Quality of MGEO Powder**

**3.2.1. Effect of Wall Material**

The effect of wall material type on the physical properties and encapsulation efficiency of MGEO powders was presented in Table 2. Moisture content can be seen from 1.24% to 1.79%, with MD- $\alpha$ -CD having the highest value (1.79%) and  $\alpha$ -CD alone showing the lowest (1.24%). Solubility can be found from 78.27% to 86.00%, and MD- $\gamma$ -CD showed the highest solubility. This result was significantly higher than that of  $\alpha$ -CD and  $\gamma$ -CD alone. These findings mean that

**Table 1:** Major sulfur volatiles of garlic essential oil (GEO) identified by gas chromatography-mass spectrometry (GC-MS)

No	Retention time (min)	Peak area*(%)	Component
9	13.094	20.42	Di-2-propenyl trisulfide
2	5.933	11.07	Diallyl disulfide
7	12.419	11.24	Methyl 2-propenyl trisulfide
21	19.866	6.4	2-Vinyl-4H-1,3-dithiine
20	19.441	5.2	3-Vinyl-1,2-dithiacyclohex-4-ene
11	14.473	5.02	Di-2-propenyl tetrasulfide
8	12.922	3.95	Allyl propyl trisulfide
15	15.977	3.74	(Z)-1-Allyl-2-(prop-1-en-1-yl) disulfane
3	7.396	3.72	Allyl methyl disulfide
18	19.159	3.14	1,2-Dithiolane

\*Peak areas were normalized to 100%

**Table 2:** Effect of encapsulation parameters on the physical properties and encapsulation efficiency of MGEO powders

Affecting factor	Moisture content (%)	Solubility (%)	Encapsulation yields (%)	Encapsulation efficiency (%)
1) Type of wall material (added GEO 2% (w/w), inlet SD temperature 160°C)				
$\alpha$ -CD	1.24±0.10 <sup>a</sup>	78.26±0.66 <sup>a</sup>	55.10±0.43 <sup>a</sup>	65.24±0.69 <sup>a</sup>
$\alpha$ -CD: MD	1.79±0.06 <sup>c</sup>	84.60±0.72 <sup>c</sup>	62.76±1.49 <sup>b</sup>	74.09±1.02 <sup>b</sup>
$\gamma$ -CD	1.67±0.03 <sup>c</sup>	82.06±0.81 <sup>b</sup>	56.05±0.21 <sup>a</sup>	66.39±0.24 <sup>a</sup>
$\gamma$ -CD: MD	1.49±0.10 <sup>b</sup>	86.00±1.00 <sup>c</sup>	79.26±0.39 <sup>c</sup>	93.68±0.12 <sup>c</sup>
2) Added GEO ratio (%) (wall material $\gamma$ -CD:MD = 1:2; inlet SD temperature 160°C)				
2	1.30±0.17 <sup>a</sup>	86.40±0.40 <sup>b</sup>	80.73±1.41 <sup>b</sup>	94.07±1.62 <sup>b</sup>
5	1.42±0.12 <sup>a</sup>	86.06±0.66 <sup>b</sup>	79.20±0.80 <sup>b</sup>	90.80±1.44 <sup>a</sup>
8	1.35±0.09 <sup>a</sup>	82.23±0.87 <sup>a</sup>	73.16±1.34 <sup>a</sup>	89.75±0.78 <sup>a</sup>
3) Inlet SD temperature (wall material $\gamma$ -CD:MD = 1:2, added GEO 5% (w/w))				
160	1.34±0.18 <sup>a</sup>	86.06±0.66 <sup>b</sup>	79.33±1.80 <sup>ab</sup>	90.40±2.11 <sup>b</sup>
170	1.22±0.15 <sup>a</sup>	86.46±0.90 <sup>b</sup>	80.53±1.28 <sup>b</sup>	91.86±1.15 <sup>b</sup>
180	1.17±0.18 <sup>a</sup>	82.80±0.60 <sup>a</sup>	76.50±1.31 <sup>a</sup>	88.93±1.62 <sup>a</sup>

<sup>a,b,c</sup>Indicates the differences between values ( $P < 0.05$ ) in column with each parameter. MGEO: Microencapsulated garlic essential oil, GEO: Garlic essential oil, MD: Maltodextrin, CD: Cyclodextrin, SD: Spray drying

combining MD with CD, especially MD and  $\gamma$ -CD, can improve the solubility and maintain moisture content at low level.

Encapsulation efficiency and yield are also affected by wall material. The MD- $\gamma$ -CD system showed the highest efficiency (93.68%) and yield (79.26%) ( $P < 0.05$ ), while  $\alpha$ -CD and  $\gamma$ -CD used individually resulted in lower values. These findings demonstrate that a combination of MD and  $\gamma$ -CD significantly improves encapsulation efficiency. This result highlights the importance of wall material combinations.

The higher encapsulation efficiency and yield observed in the MD-CD systems can be explained by the synergistic effect between the two wall materials. CD can trap volatile essential oil compounds through inclusion complex formation, while MD forms a protective film that reduces oil loss during spray drying [16,17]. In addition, MD reduces

stickiness and powder adhesion to the dryer walls, which improves powder recovery and overall process efficiency [18,19]. Therefore, the combination of MD and  $\gamma$ -CD is a suitable wall material for encapsulating GEO.

### 3.2.2. Effect of Added GEO Ratio

The results in Table 2 showed increasing the added GEO content from 2% to 8% did not affect the moisture of MGEO powders ( $P < 0.05$ ), while solubility decreased from 86.40% to 82.23%. This suggests that higher oil loads can reduce the solubility of the powder.

At 2% GEO, the encapsulation efficiency and yield were the highest (94.07% and 80.70%), but both values declined as the added GEO content increased to 8% (89.75% and 73.16%). This finding means an excessive amount of GEO negatively affects the formation of a stable microcapsule. Increasing oil content beyond an optimal level often leads to the reduction of encapsulation efficiency because of emulsion instability and insufficient wall material [19,20]. Therefore, a GEO ratio of 2–5% was considered an optimal parameter for obtaining MGEO with high solubility, encapsulation yield, and encapsulation efficiency simultaneously.

### 3.2.3. Effect of Inlet Spray Drying Temperature

It can be seen from Table 2, moisture content of MGEO powder decreased with the increasing of the inlet temperature from 160°C to 180°C. These results are similar to previous studies, which reported that water evaporation enhanced at higher spray drying temperatures [19,21]. However, at 180°C, its solubility and encapsulation yield decreased significantly, suggesting that excessive thermal stress could negatively affect the matrix and powder reconstitution properties. Encapsulation efficiency reached its highest value at 170°C but declined at 180°C, likely due to increased surface oil and partial loss of volatile compounds under rapid crust formation and thermal degradation. Therefore, an inlet temperature of 170°C was identified as optimal, providing a favorable balance between moisture content, solubility, yield, and encapsulation efficiency.

From all results at Table 2, MD- $\gamma$ -CD, 5% GEO supplement, and spray drying temperature of 170°C are suitable encapsulation parameters to balance moisture content, solubility, encapsulation yield, and encapsulation efficiency.

## 3.3. Characteristics of MGEO Powder

Based on Figure 1a, the MGEO powder appears as fine, ivory-colored particles, indicating successful matrix formation after the drying process. From visual observation, the powder is relatively homogeneous, and there are no signs of burning or discoloration, suggesting good control over drying conditions. Microscopic and SEM observations showed that the MGEO powders were solid, spherical particles with smooth or wrinkled surfaces [Figure 1b and c]. Particle size is mostly 10–20  $\mu\text{m}$ , and it is a general morphology as for spray-dried microcapsules. The FT-IR spectrum in Figure 1d displays characteristic signals at 3,407  $\text{cm}^{-1}$  (–OH, –NH), 2,925  $\text{cm}^{-1}$  (C–H), 1,649  $\text{cm}^{-1}$  (C=O, C=C), 1,160–1,024  $\text{cm}^{-1}$  (C–O–C, C–OH), and the fingerprint region at 933–525  $\text{cm}^{-1}$ . These bands confirm the presence of polysaccharides (MD), CD, and organic functional groups from GEO. Minor peak shifts and changes in intensity variation indicated inclusion and/or hydrogen-bond interactions between the GEO and the carrier matrix.

The results of TGA revealed three stages of mass-loss: (I) 30–150°C (around 6.7%, evaporation of water and residual solvents);

(II) 150–400°C (around 73%, decomposition of biopolymers and GEO); and (III) 400–600°C (around 16%, carbonaceous residue), as can be seen from Figure 1e. These results indicate that the MGEO powder remains thermally stable up to approximately 150°C, making them suitable for food and pharmaceutical applications.

XRD pattern exhibits broad and diffuse peaks, characteristic of an amorphous structure [Figure 1f]. This result indicated that the GEO is effectively encapsulated within the polymer matrix, inhibiting separate crystallization and thereby contributing to improved stability.

In summary, all FT-IR, TGA, XRD, and SEM data document the successful encapsulation of the GEO within the MD- $\gamma$ -CD system. The MGEO powder exhibits thermal stability, an amorphous structure, and microsphere particles. These properties suggest effective oil retention and potential for controlled release. Compared with garlic oil encapsulated in  $\beta$ -CD reported by Piletti *et al.*, the MGEO powder shows comparable thermal stability [22].

## 3.4. Release Behavior of GEO and Release Kinetics Modeling- Avrami Simulation

### 3.4.1. Effect of RH on Release Behavior of GEO from MGEO Powder

The effect of humidity on the release behavior was significant. At low humidity (6%), the release was very slow, reaching only 12.37% after 13 days, and the Avrami model yielding  $n \approx 0.44$  shows a Fickian diffusion-controlled mechanism. When RH increased to 40–75%, the release rate accelerated ( $k$  from 51% to 83%) while  $n$  slightly decreased (from 0.41 to 0.34), suggesting a transition toward anomalous diffusion. At 98% RH, complete release was reached within just 5 days, with a high-rate constant ( $k = 2.725$ ), which means that the release mechanism changed to rapid erosion or degradation of the matrix.

### 3.4.2. Effect of Temperature

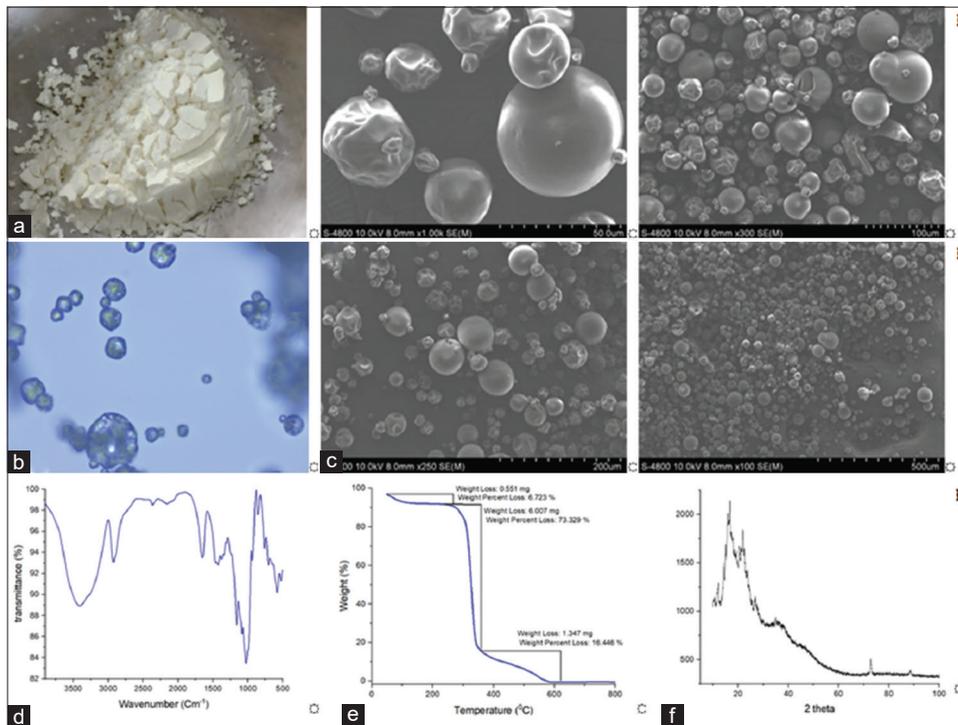
At 30°C, the release was very slow (15.73% after 35 days), a distinct characteristic of Fickian diffusion ( $n = 0.44$ ). At 65°C, the release rate increased to 23.08%, with an anomalous diffusion mechanism ( $n = 0.39$ ). At 100°C, complete release (100%) was achieved within 21 days, accompanied by a high-rate constant ( $k = 1.930$ ), indicating that the release was ruled by an erosion-controlled mechanism.

### 3.4.3. Correlation of Parameters

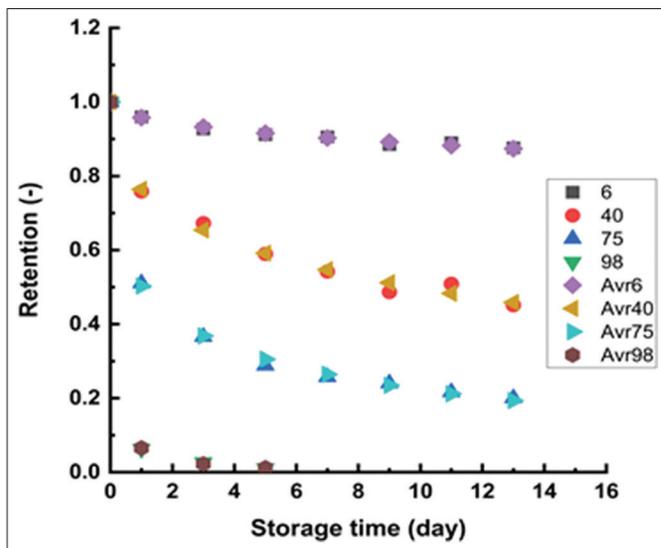
The release kinetics of GEO from MGEO powders followed the Avrami model, with  $n$  values ranging from 0.30 to 0.44, and the rate constant  $k$  strongly depending on environmental conditions. At low RH (6%) and the temperature (30°C),  $k$  remained very small (0.001–0.037) and  $n \approx 0.44$ , indicating that the release mechanism was mainly governed by Fickian diffusion through a stable CD–MD matrix. When the RH increased to 98%, or the temperature rose to 100°C,  $k$  exceeded 2.0 while  $n$  decreased to  $\sim 0.30$ , reflecting swelling and matrix erosion as the dominant release mechanism. The inclusion of  $\gamma$ -CD enhanced hydrogen bonding and strengthened the matrix, thereby slowing aroma loss and prolonging stability. These results are consistent with previous studies on spray-dried essential oil microcapsules [4].

## 3.5. Application in *Nem chua Hue*

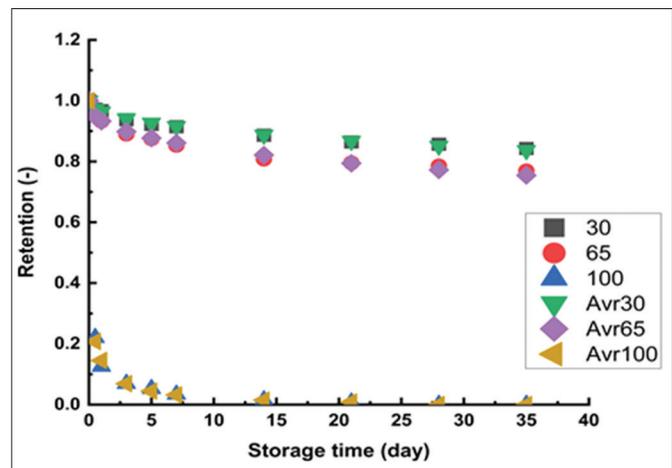
To evaluate the potential application of MGEO in the preservation of fermented pork (*Nem chua Hue*), key quality parameters, including firmness, pH, and bacterial count, were monitored over a 7-day storage period.



**Figure 1:** Characteristics of microencapsulated garlic essential oil (MGEO) powder. (a) MGEO powder, (b) Optical microscopy images, (c) Scanning electron microscopy images, (d) Fourier-transform infrared spectroscopy spectrum, (e) Thermogravimetric analysis curve, (f) X-ray diffraction pattern.



**Figure 2:** Release behavior of garlic essential oil (GEO) from microencapsulated garlic essential oil (MGEO) powder in different relative humidity (RH). 6, 40, 75, 98: Retention of GEO at RH of 6%, 40%, 75% and 98%, respectively; Avr6, Avr40, Avr75, Avr98: Retention of GEO at RH of 6%, 40%, 75%, and 98% calculated using Avrami equation, respectively.



**Figure 3:** Release behavior of garlic essential oil (GEO) from microencapsulated garlic essential oil (MGEO) powders in different temperatures. 30, 65, 100: Retention of GEO at temperatures of 30, 65, and 100°C, respectively; Avr30, Avr65, and Avr100: Retention of GEO at temperatures of 30, 65, and 100°C calculated by using Avrami equation, respectively.

The influence of MGEO on the firmness, pH, total aerobic bacteria, yeast and mold, and LAB count of *Nem chua Hue* during storage was revealed in Figures 4-6. It can be seen from Figure 4a that all displayed samples exhibited increased firmness over the storage period, reflecting the natural biochemical ripening of *Nem chua Hue*. Samples added with MGEO (0.01–0.02%) were generally firmer than the control (fresh garlic slice), particularly from days 5 to 7, indicating

the impact of the MGEO on product structure. The pH gradually decreased from approximately 6.3–6.6 on day 1 to 4.2–4.7 on day 7, consistent with lactic acid fermentation, as can be seen from Figure 4b. MGEO-supplemented samples showed a slightly slower pH decrease compared to the control, suggesting a moderated inhibition of acid-producing microorganisms. Similar results were confirmed by Nguyen *et al.*, when processing *Nem chua* by adding isolated LAB, with the pH reduced from 5.85 on day 0–4.14 on day 4 after fermentation [23].

Figure 5a-c point out all the total yeast and mold, total aerobic bacteria, and LAB concentration increased dramatically from day 1 and reached the peak on day 5, and then slightly decreased to day 7 of storage time. MGEO-supplemented samples, especially at the concentration of 0.02%, had significantly lower microbial counts than that in the control sample, indicating sustained antibacterial activity during storage. Similar trends of pH, bacterial counts were observed in the study of Nguyen *et al.* [23]. However, the time to reach the peak of indicators in this study was slower than that reported by Nguyen *et al.*, at 5 days rather than 2 days after processing [23].

The incorporation of MGEO in *Nem chua Hue* contributed to the product quality characteristics by maintaining firmness, adjusting pH reduction, and limiting aerobic bacterial growth. These findings highlight the potential application of MGEO as a natural preservative, contributing to extended shelf-life and improved microbiological safety.

Antibacterial efficacy increased with the increase of MGEO concentration (0.01% <0.015% <0.02%), confirming significant microbial inhibition during storage period. All MGEO-supplemented samples illustrated a slower pH reduction compared to the control

sample, helping preserve product structure. The firmness of MGEO-supplemented samples in Figure 4a indicated effective maintenance of texture. Therefore, adding 0.015% MGEO balanced microbiological safety, sensory quality, and extended shelf-life.

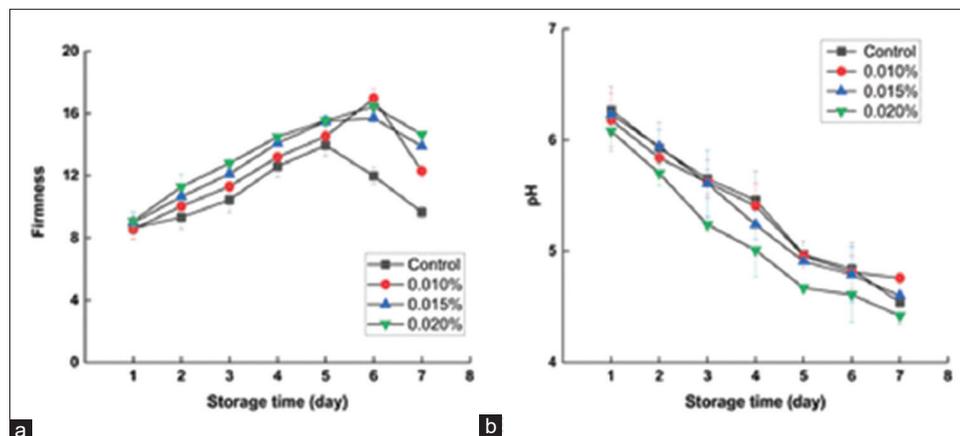
The impact of MGEO on LAB count is a key factor because LAB are responsible for the acidification and flavor modification of *Nem chua Hue*. In the present study, MGEO caused only a moderate decrease in LAB counts. However, the fermentation process was not negatively affected, as the pH still decreased to the desired range of 4.5–5.0 within the fermentation time. This result agreed with previous studies showing that LAB are more tolerant to garlic-derived organosulfur compounds than spoilage or pathogenic microorganisms. Allicin in garlic mainly reacts with thiol (–SH) groups of microbial enzymes, which can inhibit cell metabolism [24]. At the applied concentration (0.015%), MGEO appeared to selectively suppress undesirable microorganisms, including aerobic bacteria, molds, and yeasts, while allowing LAB to remain active.

Table 3 presents the sensory scores of *Nem chua Hue* supplemented with MGEO at different concentrations. Color scores were not significantly different by the treatments, whereas odor, taste, and texture varied depending on the oil content. The sample containing 0.01% MGEO achieved the highest overall acceptability index (Overall acceptance index [OAI] = 6.80), reflecting positive perceptions of aroma and taste. At 0.02% MGEO, the OAI decreased to 5.98 due to the strong garlic odor, which limited consumer acceptance. The 0.015% MGEO treatment provided the most balanced outcome (OAI = 6.63), combining acceptable sensory quality with prolonged microbiological stability. Overall, 0.015% MGEO was identified as the optimal level, achieving a balance between sensory acceptability, microbial safety,

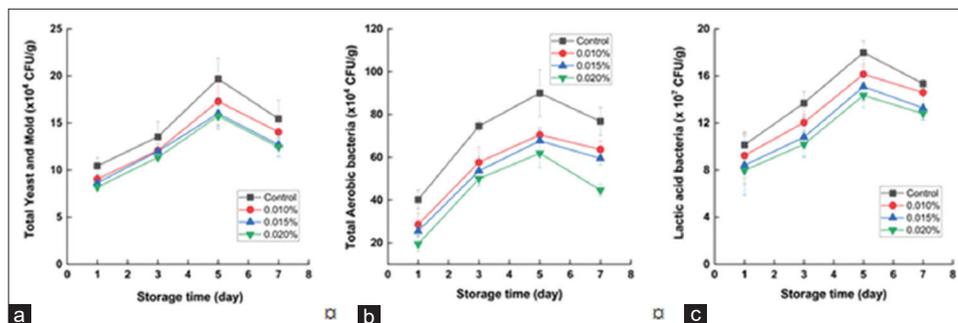
**Table 3:** Sensory attributes (day 7) and OAI

Treatment	Color	Odor	Taste	Texture	OAI
Control	5.9 <sup>a</sup> ±1.39	5.1 <sup>b</sup> ±1.25	5.1 <sup>b</sup> ±1.10	6.3 <sup>b</sup> ±0.70	5.60
0.010% GEO	6.3 <sup>a</sup> ±1.28	7.1 <sup>a</sup> ±1.28	7.3 <sup>a</sup> ±1.10	6.5 <sup>b</sup> ±0.95	6.80
0.015% GEO	6.3 <sup>a</sup> ±0.80	6.7 <sup>a</sup> ±0.92	6.4 <sup>ab</sup> ±1.06	7.1 <sup>a</sup> ±1.08	6.63
0.020% GEO	6.2 <sup>a</sup> ±1.40	5.4 <sup>b</sup> ±1.35	4.9 <sup>b</sup> ±0.97	7.4 <sup>a</sup> ±1.20	5.98

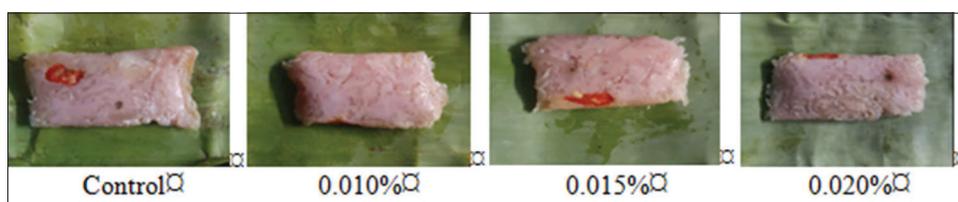
OAI: (Colour+Aroma+Taste+Texture)/4. GEO: Garlic essential oil, OAI: Overall acceptance index



**Figure 4:** Effect of added microencapsulated garlic essential oil powder on *Nem chua Hue* characteristics. (a) Firmness, (b) pH.



**Figure 5:** Bacteria growth during storage time of *Nem chua Hue*. (a) Total yeast and molds, (b) Total aerobics bacteria, (c) Lactic acid bacteria.



**Figure 6:** *Nem chua Hue* after 5 days storage.

and storage stability, whereas 0.01% was preferred when flavor was prioritized and 0.02% when maximum antibacterial protection was concerned. The sensory results revealed that the addition of MGEO did not significantly reduce overall acceptability. This confirms that microencapsulation effectively controlled the release of volatile compounds, preventing excessive aroma intensity while preserving the characteristic sensory profile of *Nem chua Hue*.

From an application perspective, *Nem chua Hue* is commonly wrapped by banana leaves, fermented at room temperature over a few days, and stored under this condition where moisture availability is high. The Avrami results indicated that at high RH the GEO is completely released from MGEO within 5 days. Therefore, MGEO can provide a more sustained delivery of organosulfur compounds during fermentation and early storage, supporting inhibition of undesirable microorganisms, including aerobic bacteria, molds, and yeasts. This linkage helps explain the observed microbial reductions and the extension of *Nem chua Hue*'s shelf-life. Ultimately, MGEO offers a practical strategy to enhance microbial stability of *Nem chua Hue* without compromising sensory quality or traditional characteristics.

Although this study was mainly concerned with technological optimization and practical application rather than detailed antimicrobial mechanism elucidation, the sustained release of organosulfur compounds from MGEO can be responsible for the observed reduction in total aerobic bacteria and delayed acidification in *Nem chua Hue*. DATS and DADS, which are abundant in GEO, are widely reported to prevent microbial growth by disrupting cell membranes and interfering with enzymatic activity. In order to stabilize these volatile compounds and enable gradual release under fermentation and storage conditions, it is crucial to apply the MD- $\gamma$ -CD encapsulation matrix. As a result, MGEO prevented excessive suppression of LAB necessary for fermentation while offering a more consistent antimicrobial action as compared to fresh garlic slices.

MGEO can be used as a natural preservative in fermented meat products since similar controlled-release benefits of encapsulated essential oils have been reported in food preservation systems.

#### 4. CONCLUSION

With an encapsulation efficiency of around 92%, the MD- $\gamma$ -CD combination produced stable microcapsules with effective regulated release of GEO.

With  $n \approx 0.30$ – $0.44$ , the release kinetics followed the Avrami model, indicating primarily Fickian diffusion under low RH conditions and a shift to erosion-controlled release at higher RH levels. This provided a logical basis for selecting storage and release conditions.

Supplementing *Nem chua Hue* with 0.01–0.02% MGEO considerably decreased total aerobics bacteria, yeasts, and molds, reduced acidification, and maintained LAB and product firmness. Among the tested levels, the highest microbiological safety was achieved at 0.02%

MGEO, while the most balance between sensory acceptability and microbial control was at 0.015%.

In summary, this study presents an optimal set of encapsulating parameters (wall material MD- $\gamma$ -CD, 5% GEO, inlet temperature 170°C) and recommended dosage of 0.015% MGEO for practical implementation to extend the shelf-life of *Nem chua Hue* without losing sensory acceptance.

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#### 6. CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

Nguyen Thi Van Anh: Conceptualization, Methodology, Investigation, Project administration, Writing - original draft, Funding acquisition. Tran Viet Tai Duc: Investigation, Visualization. Nguyen Duc Chung: Conceptualization, Methodology. Doan Thi Thanh Thao: Methodology, Investigation. Phan Thanh Long: Conceptualization, Methodology. Nguyen Quoc Thang: Conceptualization, Methodology, Validation. Le Van Tan: Funding acquisition, Supervision, Project administration, Writing - review and editing (Corresponding author). All authors have read and agreed to the published version of the manuscript.

Main authors: Nguyen Thi Van Anh (First author), Le Van Tan (Corresponding author).

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#### 8. CONFLICTS OF INTEREST

The authors report no financial or any other conflicts of interest in this work.

#### 9. ETHICAL APPROVALS

This study does not involve human or animal subjects.

#### 10. DATA AVAILABILITY

All data from the current study are available from the corresponding author upon request.

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