

# Preparation of shallot capsules with variations in the ratio and type of encapsulating materials

[Produksi kapsul bawang merah dengan variasi rasio dan jenis bahan enkapsulan]

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## ABSTRACT

*Shallots (*Allium ascalonicum*) are a perishable spice crop prone to rapid degradation. One method to extend their shelf life is through encapsulation. Shallot capsules possess the potential to mitigate post-harvest losses and improve the utility of shallots. The encapsulating material and its proportion significantly influence the quality of the capsules. This study employed coacervation techniques using alginate combined with oxidized starch as encapsulating materials. The objective of this research was to evaluate the effect of encapsulant-to-shallot ratios and the influence of different combinations of encapsulants on the quality of shallot capsules. The research was conducted using a Completely Randomized Design (CRD) with a factorial arrangement consisting of two factors and carried out in 3 repetitions. The first factor was the ratio of alginate and photo-oxidized starch, and the second factor was the ratio of encapsulating materials to shallot powder. The research involved the preparation of oxidized starch, shallot powder, and shallot capsules, followed by characterization of the capsules. The evaluated parameters included yield, moisture content, total polyphenol content, encapsulation efficiency, loading capacity, SEM analysis, FTIR analysis, solubility, and a triangle test. Data were analyzed using ANOVA at a significance level of  $\leq 5\%$  and the triangle test was analyzed using a one-tailed binomial table ( $p = 0.001$ ). The results showed that the treatments had a significant effect on yield, moisture content, total polyphenol content, encapsulation efficiency, loading capacity, and solubility. Additionally, the triangle test revealed a statistically significant difference between the two treatments at the 0.1% significance level.*

*Keywords: alginate, coacervation technique, oxidized starch, shallot*

## ABSTRAK

Bawang merah (*Allium ascalonicum*) merupakan tanaman rempah yang mudah mengalami kerusakan. Upaya penanganannya dengan pengolahan menjadi kapsul bawang merah. Kapsul bawang merah berpotensi mengurangi kehilangan pasca panen dan meningkatkan kegunaan bawang merah. Namun, kualitas kapsul sangat bergantung pada bahan pembungkus dan rasionya. Metode yang digunakan yaitu teknik *coacervation* yang menggunakan bahan pengkapsul alginat dikombinasikan dengan pati terfotooksidasi. Tujuan penelitian ini yaitu untuk mengetahui pengaruh perbandingan bahan pengkapsul dengan bawang merah dan pengaruh rasio kombinasi bahan pengkapsul yang digunakan terhadap kualitas kapsul bawang merah. Penelitian ini dilakukan dengan menggunakan Rancangan Acak Lengkap (RAL) yang terdiri dari dua faktor dan dilakukan sebanyak 3 kali ulangan. Faktor pertama adalah perbandingan alginat dan pati fotooksidasi dan faktor kedua adalah perbandingan bahan enkapsulasi dengan tepung bawang merah. Penelitian ini terdiri dari pembuatan pati terfotooksidasi, pembuatan bubuk bawang merah, pembuatan kapsul bawang merah, dan pengujian karakteristik kapsul bawang merah. Parameter penelitian yang dilakukan yaitu rendemen, kadar air, total polifenol, efisiensi enkapsulasi, *loading capacity*, SEM, FTIR, kelarutan, dan uji perbedaan segitiga. Analisis data menggunakan uji ANOVA dengan taraf nyata  $\leq 5\%$ . Analisis perbedaan segitiga menggunakan tabel binomial one-tailed  $p=0,001$ . Hasil penelitian menunjukkan bahwa perlakuan yang digunakan berpengaruh nyata terhadap rendemen, kadar air, total polifenol, efisiensi enkapsulasi, *loading capacity*, dan kelarutan. Pada parameter uji perbedaan segitiga kedua perlakuan tersebut juga menunjukkan hasil berbeda nyata pada taraf uji 0,1%.

Kata kunci: alginat, bawang merah, pati terfotooksidasi, teknik *coacervation*

## Introduction

Shallots (*Allium ascalonicum*) are extensively utilized as both a culinary spice and a medicinal ingredient. Data from the Indonesian Central Statistics Agency (BPS, 2024) indicated that shallot production in Indonesia increased significantly from 2022 to 2023. However, post-harvest handling remains a challenge, with physical damage and weight loss often leading to price fluctuations. Improper post-harvest management can result in losses of up to 20–40%, a problem worsened by the perishable nature of shallots and their short shelf life (Ayu et al., 2023). One of the post-harvest handling alternatives for shallots is processing them into shallot capsules, which helps minimize potential spoilage.

Shallots have significant potential as an instant seasoning to enhance the flavor and aroma of dishes. The purpose of instant seasoning, besides extending shelf life and enhancing durability, is to offer practical use (Rahmadhani et al., 2021), as well as to facilitate distribution and storage. The aroma of shallots can be preserved using encapsulation techniques. One such method, the coacervation encapsulation process, involves the separation of a colloidal system into two liquid phases, forming a dense polymer-rich phase (the coacervate) that encapsulates the active material (Napiórkowska et al., 2022). This method is valued for its efficiency, ease of implementation, and high encapsulation efficiency (Fitriyani et al., 2023). Compared to other encapsulation techniques, the coacervation method stands out for its benefits, including high coating activity (99%) and stability under conditions of high humidity and temperature (Gürbüz et al., 2020). Alginate, as an encapsulating material, is capable of forming gels through ionic interactions with calcium ions, such as calcium chloride, resulting in a stable blend that is suitable for encapsulation applications (Bustos et al., 2023). However, calcium alginate capsules are highly porous (Massana-Roquero et al., 2021), necessitating the use of additional encapsulating materials, such as photo-oxidized starch, which has micro-sized pores that can seal the pores of alginate.

In a study conducted by Palupi et al. (2014), red chili was encapsulated using the coacervation technique with alginate and photo-oxidized tapioca as encapsulating materials, with suspension concentrations of 5% and 10%. The encapsulation efficiency for samples with a 50% substitution of photo-oxidized tapioca at a 10% suspension concentration was found to be the highest. This is because the smaller pore size of photo-oxidized tapioca compared to alginate can seal the larger pores of alginate, thus retaining more of the capsaicin content in red chili. Building on this concept, encapsulation techniques have also shown promise in addressing challenges in other agricultural products, such as shallots.

Shallot capsules hold the potential for reducing post-harvest losses and enhancing the usability of shallots; however, the quality of the capsules is highly dependent on the encapsulating material and its ratio (Bustos et al., 2023). A lack of understanding of the appropriate concentration and combination of encapsulating materials often leads to poor capsule stability, low efficiency, and compromised quality. To address this issue, this study aimed to determine the effect of the encapsulating material ratio on shallots, as well as the impact of the encapsulating material combination ratio on the quality of shallot capsules. Additionally, the study sought to establish the optimal concentration and combination ratio of encapsulating materials that produce high-quality shallot capsules.

## Materials and methods

### Materials and equipment

The materials used for the preparation of shallot powder capsules were shallots obtained from the Traditional Market in Jember, alginate, commercial corn starch (Xingmao Corn Starch), 30% H<sub>2</sub>O<sub>2</sub> (technical grade), distilled water, and aluminum foil. The materials required for the analysis procedure were distilled water, Folin-Ciocalteu reagent (Merck), gallic acid (Merck), sodium carbonate, filter paper, coffee powder (Kapal Api), and fried shallots (B.J. brand).

The equipment used for the preparation of shallot powder capsules included a blender (Miyako BL-152 PF AP), analytical balance (Ohaus Ap-310-O, Switzerland), syringe, beaker glass, hot plate, thermometer, spatula, oven, knife, cutting board, baking tray, a set of photo-oxidation equipment with a UV-C lamp (Germilite) and pump (MOSSWELL model DB-125), drying cabinet, and magnetic stirrer (Vitlab). The equipment required for the analysis procedure included a moisture analyzer, pi-pump, spectrophotometer, hot plate, magnetic stirrer, spatula, analytical balance, spray bottle, desiccator, oven, centrifuge, SEM testing equipment, FTIR testing equipment, and glassware.

### **Research methods**

This research consisted of four stages: the preparation of photo-oxidized starch, the preparation of shallot powder, the preparation of shallot powder capsules, and the analyses of shallot powder capsules. The study was conducted using a Completely Randomized Design (CRD) with a factorial arrangement, consisting of two factors. The first factor was the ratio of alginate and photo-oxidized starch as the encapsulating materials (A), which included: A100 (100:0), A75 (75:25), A50 (50:50), and A25 (25:75). The second factor was the ratio of encapsulating materials to shallot powder (K), which consisted of 1:2 (K2) and 1:3 (K3). The experiment was repeated 3 times, resulting in 24 samples. The formulation of the encapsulating material treatments used in the experiment can be seen in Table 1.

**Table 1.** Encapsulation material formulation

Alginate : Photooxidized Starch Ratio (A)	Shallot : Encapsulating Material (K)	
	1:2	1:3
100:0	A100K2	A100K3
75:25	A75K2	A75K3
50:50	A50K2	A50K3
25:75	A25K2	A25K3

### **Preparation of photo-oxidized starch**

The preparation of photo-oxidized starch was based on modifications from the research conducted by Palupi et al. (2020), using the photo-oxidation technique with UV-C light exposure and a strong oxidizer, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). The first step in the starch photo-oxidation technique involved preparing 4000 ml of suspension, consisting of 200 g starch (5% w/v), 1% (v/v) or 133.33 ml of 30% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), and 3666.67 ml of distilled water. The ingredients were homogenized and then placed into the sample tube of the photo-oxidation apparatus, which was equipped with a stirrer. Photo-oxidation was conducted at room temperature through a circulation system and a sample tube equipped with a jacket tank filled with cold water to prevent a temperature increase due to UV-C lamp energy. The suspension was pumped through the UV-C lamp (UV-C irradiation). The exposure was initiated by pressing the power button on the UV-C lamp of the photo-oxidation apparatus. The photo-oxidation process was carried out for 1 hour. The photo-oxidized suspension was then drained from the sample tube through the output tap and subsequently dried in a drying cabinet at 50°C for 24 hours. After the drying process, the resulting starch microparticles, still in aggregate form, were broken down into fine starch microparticles.

### **Preparation of shallot powder**

The preparation of shallot powder was conducted based on modifications from the research of Dewayani et al. (2020). The process began with peeling the shallots, followed by washing them. The cleaned shallots were then thinly sliced to facilitate the drying process. The shallots were dried using an oven at 60°C for 20 hours. Once dried, the shallots were blended into a fine powder and stored in a sealed container.

### ***Preparation of shallot capsules using the coacervation technique***

The preparation of shallot capsules was carried out based on modifications from the research of Palupi et al. (2014). Stage 1 of the shallot capsule preparation using the coacervation technique began by pouring 100 ml of distilled water into a beaker glass, followed by stirring for 5 minutes at 50°C. Micro-particle starch was then added, and the mixture was stirred again for 30 minutes at 75°C. The homogeneous solution was left to stand for 30 minutes at room temperature. Afterward, the solution was stirred, and alginate was added, followed by shallot powder. Once all ingredients were mixed, the mixture was drawn into a syringe and proceeded to Stage 2.

In the coacervation method, 11.1 grams of  $\text{CaCl}_2$  were dissolved in 100 mL of distilled water. Once the  $\text{CaCl}_2$  was fully dissolved, the suspension was dropped into the  $\text{CaCl}_2$  solution using a syringe. The formed beads were left in the  $\text{CaCl}_2$  solution and stirred for 10 minutes. Afterward, the beads were filtered and placed on a baking tray lined with aluminum foil. The drying process was conducted at 50°C for 20 hours. The analyses performed on the shallot powder capsules included yield, moisture content, total polyphenol content, encapsulation efficiency, loading capacity, SEM, FTIR, solubility, and a triangle difference test.

### ***Yield***

The procedure for yield analysis was adapted from Tanuwidjaja & Prasetyanto (2022). The yield was calculated based on the weight of the shallot capsules produced from the weight of the materials used. The encapsulated shallot yield was calculated using the formula:

$$\text{Yield (\%)} = \frac{\text{Capsule weight} - (\text{Capsule moisture content} \times \text{capsule weight})}{\text{Dry material weight}} \times 100$$

Capsule weight refers to the total weight of the encapsulated shallot. Dry material weight represents the initial material weight of photo-oxidized starch, alginate, and shallot powder without distilled water.

### ***Moisture content***

The moisture content was tested using a moisture analyzer adapted from Nurhidayati (2021). The principle of this test is the weight loss during heating at 130°C, which is considered the moisture content of the sample. An aluminum dish was placed in the moisture analyzer, and 1 gram of the sample was added. The lid was closed, and the initial weight was recorded. Heating took 3-5 minutes, and the test was complete when a sound was heard. The final weight and moisture content displayed on the screen were recorded.

### ***Total polyphenols***

The polyphenol content analysis was adapted from Sujana et al. (2022) and conducted using the Folin-Ciocalteu method. A 2 mg/ml sample was homogenized in distilled water using a vortex for 5 minutes. A 0.2 ml sample solution was placed in a test tube, and 0.8 ml of diluted Folin-Ciocalteu reagent (1:10 v/v) was added. The mixture was left to stand for 20 minutes. Then, 4 ml of  $\text{Na}_2\text{CO}_3$  (8.5% w/v) was added, homogenized, and left to stand for 90 minutes in a dark place. A gallic acid standard curve was prepared using concentrations of 0, 50, 100, 150, 200, and 250  $\mu\text{l}$ . The absorbance was measured at 765 nm. The total polyphenol content of the sample was calculated based on the gallic acid standard curve and expressed as mg/g sample.

$$\text{Total polyphenol (mg GAE/g)} = \frac{X \times \text{dilution factor} \times \text{volume (ml)}}{\text{Sample weight (g)}}$$

### **Encapsulation efficiency**

Encapsulation efficiency (EE) was adapted from Rosales et al. (2021) and calculated based on the encapsulated polyphenol content (AE) and the total polyphenol content of the capsule (AT) with ethanol as the solvent. The encapsulation efficiency (%) was calculated using the formula:

$$\text{Encapsulation efficiency (EE)} = \frac{\text{Encapsulated polyphenol content (A}_E\text{)}}{\text{Total polyphenol content (A}_T\text{)}} \times 100\%$$

### **Loading capacity**

Procedure for loading capacity was adapted from Rosales et al. (2021). The loading capacity represents the polyphenol content of the shallot powder (AE) per unit weight of powder (W). The loading capacity (L) was calculated using the formula:

$$\text{Loading capacity (L)} = \frac{\text{Polyphenol content (A}_E\text{)}}{\text{Powder weight (W)}} \times 100\%$$

### **Particle morphology**

The particle morphology analysis was tested using Scanning Electron Microscopy (SEM) (Hitachi TM3000 Tabletop Microscope) following the method of Palupi et al. (2020). The sample was attached to conductive tape using a brush, and the area around the tape was cleaned using compressed air. The sample was then observed at magnifications of 400× and 1000×.

### **Functional group**

FTIR analysis was adapted from Damto et al. (2023) and conducted to identify the chemical bonds present in the shallot capsule samples. The chemical bonds were indicated by different peaks in the FTIR spectrum. Fourier Transform Infrared (FTIR) spectroscopy involves transmitting light through the sample and measuring the intensity of the light with a detector. The infrared spectrum was plotted as intensity versus energy, wavelength (μm), or wavenumber (cm<sup>-1</sup>).

### **Solubility**

Solubility testing was adapted from Hasan et al. (2022) and performed by weighing 1 gram of the sample, dissolving it in 10 ml of distilled water, and homogenizing it until fully dissolved. The solution was then centrifuged at 3000 rpm for 10 minutes. The supernatant was placed in a weighing bottle and dried in an oven at 105°C for 24 hours, after which the weight was measured until constant. Solubility in water was calculated using the formula:

$$\text{Solubility (\%)} = \frac{\text{Dry supernatan weight}}{\text{Sample weight}} \times 100\%$$

### **Aroma difference**

The triangle difference test was used to determine sensory differences in aroma between shallot capsules and fried shallots, following the method adapted from Rodriguez (2021). The shallot capsule sample tested was A50K3, and the test involved 30 panelists. Each panelist was presented with three samples simultaneously, two of which were identical. To minimize bias, the samples were arranged in three random combinations and labeled with randomly assigned three-digit codes. Panelists were instructed to smell the samples from left to right and identify the one that differed from the others. To neutralize their sense of smell between tests, they were asked to smell coffee powder.



## Results and discussion

### Yield

The results of statistical analysis using ANOVA at a significance level of  $\alpha \leq 0.05$  showed that the treatment of encapsulating material ratios (alginate and photo-oxidized starch) and the ratio of encapsulating material to shallots in the production of shallot capsules had a significant effect on the resulting yield. The yield values of the shallot capsule samples are shown in Figure 1.

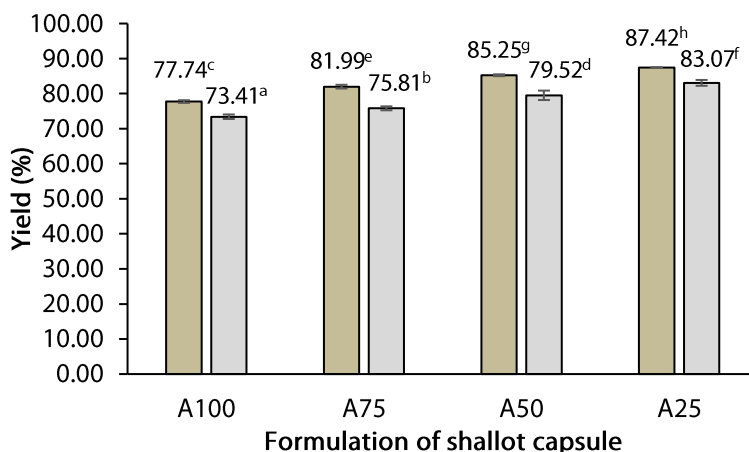


Figure 1. Yield of shallot capsules from different ratios of shallot to encapsulant (■ 1:2 and □ 1:3)

Based on the results obtained, the higher the addition of photo-oxidized starch, the greater the yield produced. This is because photo-oxidized starch has smaller pores, compared to alginate. In the encapsulation process, the larger pores of alginate are covered by the smaller pores of photo-oxidized starch, so a higher addition of photo-oxidized starch can retain more material, resulting in a higher yield (Tian et al., 2022). Furthermore, the shallot and encapsulant ratio of 1:3 shows a higher yield compared to the ratio of 1:2. This is due to the increased amount of encapsulating material used, which contributes to a greater amount of solid content produced (Kurniasari et al., 2022).

### Moisture content

The results of statistical analysis using ANOVA at a significance level of  $\alpha \leq 0.05$  showed that the treatment of encapsulating material ratios (alginate and photo-oxidized starch) and the ratio of encapsulating material to shallots in the production of shallot capsules had a significant effect on the resulting moisture content. The moisture content values of the samples are shown in Figure 2.

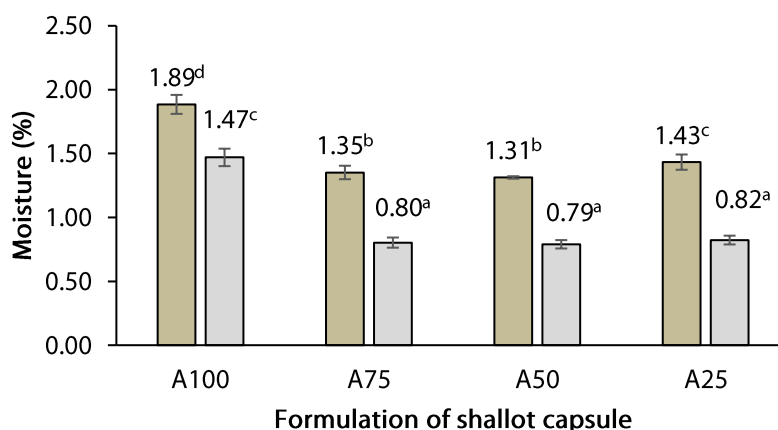
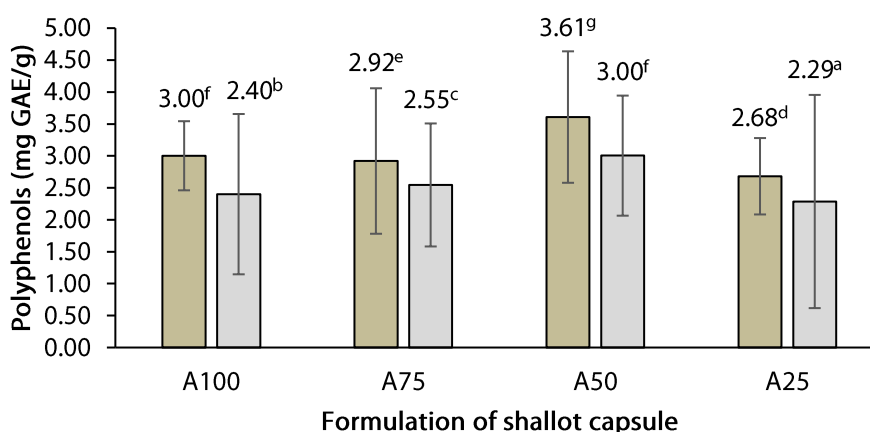


Figure 2. Moisture content of shallot capsules from different ratios of shallot to encapsulant (■ 1:2 and □ 1:3)

The moisture content of the shallot and encapsulant ratio of 1:3 was higher compared to the ratio of 1:2 due to the increased amount of encapsulating material. Additionally, a higher proportion of alginate (A100) in the encapsulant resulted in higher moisture content, likely due to the hygroscopic nature of alginate, which enables it to absorb moisture from the environment (Eslami et al., 2023). This aligns with Zhang et al. (2022), stating that alginate, as a natural linear polysaccharide, possesses high hygroscopicity.

### Total polyphenols

The total polyphenols in this study refer to the polyphenol content in shallot capsules. The results of statistical analysis using ANOVA at a significance level of  $\alpha \leq 0.05$  showed that the treatment of encapsulating material ratios (alginate and photo-oxidized starch) and the ratio of encapsulating material to shallots in the production of shallot capsules had a significant effect on the resulting polyphenol content. The total polyphenol values are shown in Figure 3.



**Figure 3.** Polyphenol content of shallot capsules from different ratios of shallot to encapsulant (■ 1:2 and □ 1:3)

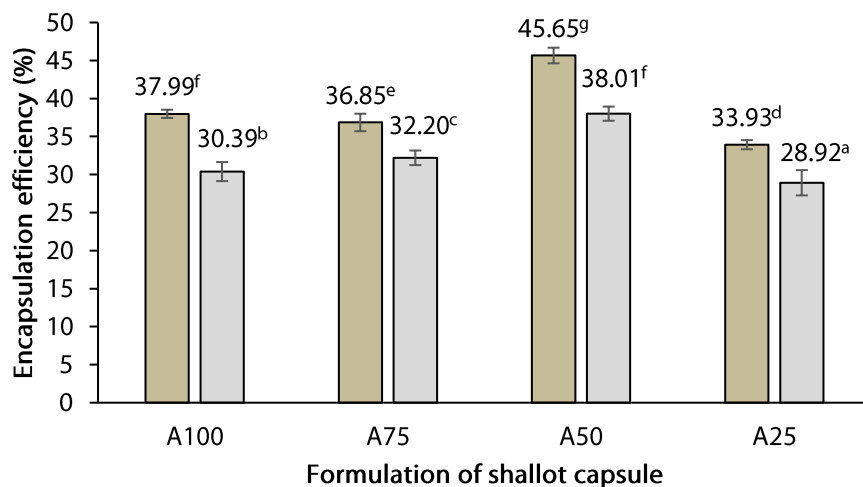
Results indicate that a higher encapsulant ratio (1:3) corresponds to increased polyphenol content. This aligns with the findings of Yadav et al. (2019), who reported that microcapsules with higher coating concentrations showed greater encapsulation efficiency and more controlled release of phenolic compounds. These results suggest that higher coating concentrations contribute to thicker encapsulation layers, enabling more controlled release and better preservation of polyphenol content. Additionally, increasing the amount of photo-oxidized starch in the shallot capsules led to higher polyphenol content, likely due to the pore size characteristics of the encapsulating materials. Alginate has larger pores compared to photo-oxidized starch. Oxidized starch has micro-sized pores in the range of 19.18–48.82  $\mu\text{m}$  (Okekunle et al., 2020). This supports Yadav et al. (2019), where encapsulants with higher efficiency and controlled release also showed smaller particle sizes.

The sample with the lowest polyphenol content was A25K2, with a value of 2.286 mg GAE/g. In the A25K2 sample, which contained 75% photo-oxidized starch and 25% alginate, the low polyphenol content may be attributed to the ratio being insufficient to form an effective encapsulation matrix. This is consistent with the study by Palupi et al. (2014), where a 50% concentration of photo-oxidized tapioca and alginate could not retain capsaicin effectively due to the inability to form a proper encapsulation matrix.

### Encapsulation efficiency

Encapsulation Efficiency (EE) is a measure of how effectively an active substance is incorporated within a carrier system such as nanoparticles or microcapsules (Soltanzadeh et al., 2021). It is defined as the ratio of the amount of active substance encapsulated (or trapped) within the carrier to the total amount of active substance initially used in the formulation. The polyphenol content in the sample affects the encapsulation efficiency value. The results of statistical analysis using ANOVA at a significance level of  $\alpha \leq 0.05$  showed

that the treatment of encapsulating material ratios (alginate and photo-oxidized starch) and the ratio of encapsulating material to shallots in the production of shallot capsules had a significant effect on the resulting encapsulation efficiency. The encapsulation efficiency values are shown in Figure 4.

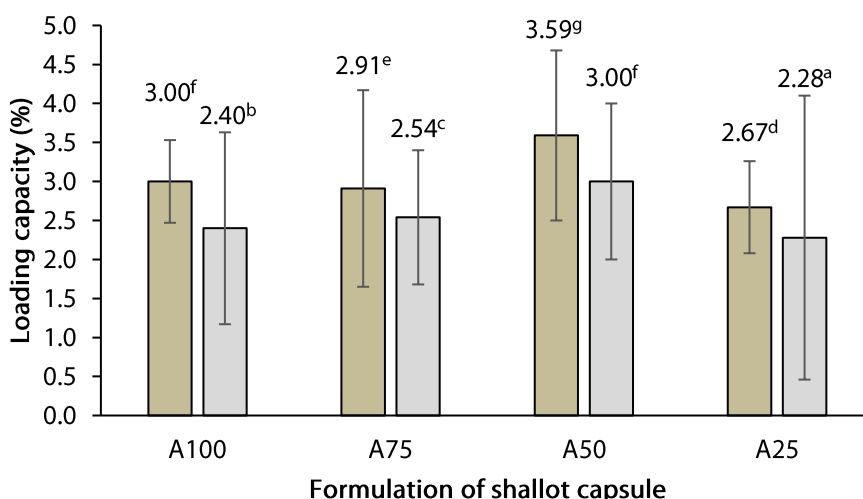


**Figure 4.** Encapsulation efficiency of shallot capsules from different ratios of shallot to encapsulant (■ 1:2 and □ 1:3)

Based on the results obtained, encapsulation efficiency values also increased with higher additions of photo-oxidized starch. The highest encapsulation efficiency was achieved at an encapsulant ratio of 1:3, reaching 45.654%. This is because modified starches are frequently used in encapsulation processes due to their ability to form dense interfacial coatings. According to Zabot et al. (2022), these coatings effectively protect and bind the encapsulated compounds, enhancing both encapsulation efficiency and the stability of bioactive ingredients during storage.

#### **Loading capacity**

The results of statistical analysis using ANOVA at a significance level of  $\alpha \leq 0.05$  showed that the treatment of encapsulating material ratios (alginate and photo-oxidized starch) and the ratio of encapsulating material to shallots in the production of shallot capsules had a significant effect on the resulting loading capacity. The loading capacity values are shown in Figure 5.



**Figure 5.** Loading capacity of shallot capsules from different ratios of shallot to encapsulant (■ 1:2 and □ 1:3)

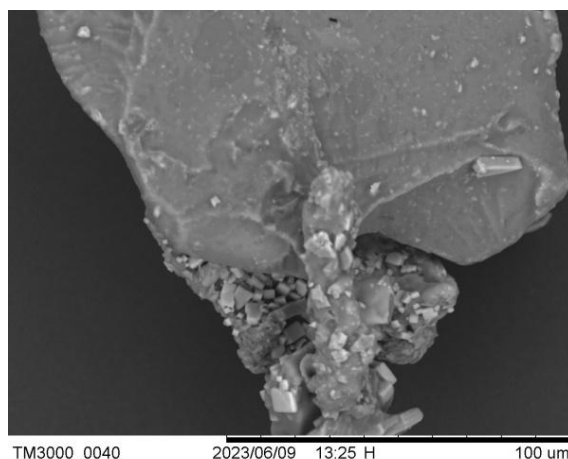
The loading capacity at the shallot: encapsulant ratio of 1:3 was higher than that at the ratio of 1:2, reaching 3.59%. Increasing the amount of encapsulant added results in a higher loading capacity. According to Feby et al. (2024), the loading capacity value represents the amount of material that can be



encapsulated within a given number of microcapsules. Additionally, a higher amount of photo-oxidized starch in the formulation correlates with an increase in loading capacity. This is likely due to the small pore size of photo-oxidized starch (Okekunle et al., 2020), compared to alginate. Encapsulants with larger pores allow more compounds to escape, thereby reducing the overall loading capacity. The loading capacity of sample A25K2 was the lowest, which may be attributed to its lower polyphenol content.

### **Particle morphology**

The particle morphology of the samples was examined using Scanning Electron Microscopy (SEM). The sample used in the SEM test was A50K3, chosen because it had the highest polyphenol content. The SEM test results for the A50K3 sample are shown in Figure 6.



**Figure 6.** SEM test results for sample A50K3 at 1000x magnification

Figure 6 presents the SEM micrograph of sample A50K3 at 1000x magnification, revealing a dense distribution of small white granules characteristic of alginate-based matrices. The surface texture of the alginate-based matrix also appeared rough, with visible cracks and wrinkles, consistent with previous findings on alginate bead morphology (Li et al., 2022). The surface morphology further exhibits irregular, compacted granules forming clumps, likely due to the heating process during the preparation of the shallot capsules, where starch gelatinization occurred in the presence of water. In the SEM micrograph, the shallot powder was presumed to have merged with the starch. Moreover, the presence of photo-oxidized starch is evident from its rough, irregular texture. This observation aligns with previous studies indicating that oxidation induces molecular degradation, leading to an increased surface area and the formation of a more porous, irregular structure (Aaliya et al., 2022).

### **Functional group analysis**

FTIR characterization was conducted to identify the functional groups present in the materials. FTIR analysis was performed on alginate, photo-oxidized starch, shallot powder, and sample A50K3, which was selected due to its highest polyphenol content. The FTIR test results are shown in Figure 7. The functional group analysis using FTIR of sample A50K3 showed the presence of carboxylic acid dimers, as evidenced by characteristic absorption in the wavenumber range of  $1300\text{--}1280\text{ cm}^{-1}$ , which is typically attributed to the C–O stretching vibration of carboxyl groups (Smith, 2018). This finding is consistent with the known chemical structure of alginate, which contains carboxyl functional groups that can engage in intermolecular hydrogen bonding. In addition, shallot powder exhibited absorption in the  $1730\text{--}1750\text{ cm}^{-1}$  range, corresponding to ketone-dialkyl bonds (Nandiyanto et al., 2019), which are indicative of the presence of carbonyl-containing compounds such as flavonoids and sulfur compounds commonly found in shallots (Indrasari et al., 2021). Moreover, the FTIR spectrum of photo-oxidized starch displayed strong absorption in the  $1740\text{--}1700\text{ cm}^{-1}$  range, which corresponds to aldehyde-aromatic bonds arising from

oxidative degradation of starch molecules (Nandiyanto et al., 2019). This transformation typically results from the breakdown of glycosidic linkages, leading to the formation of low-molecular-weight carbonyl compounds. These types of bonds were also present in sample A50K3 at the same wavenumber ranges, confirming the incorporation of key functional groups from its raw materials.

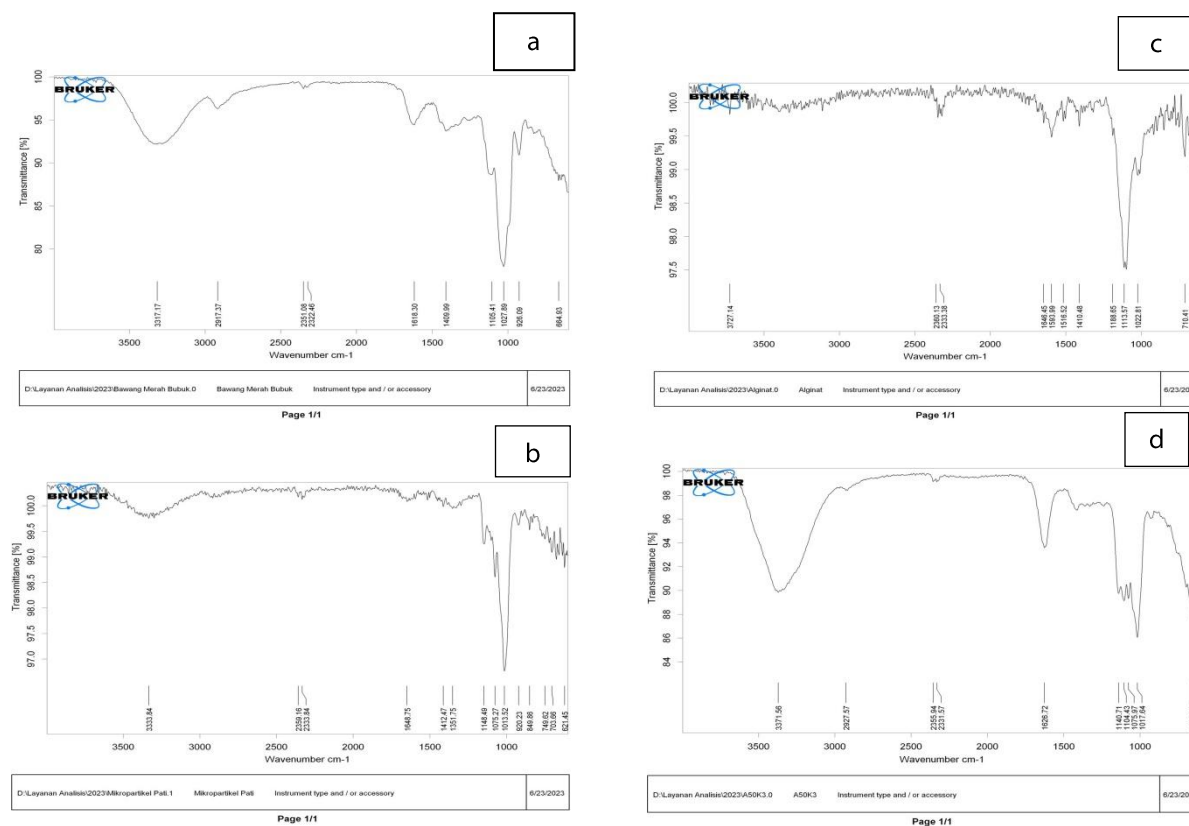


Figure 7. FTIR spectrum of (a) shallot powder, (b) starch microparticles, (c) alginate, and (d) sample A50K3

## Solubility

The results of statistical analysis using ANOVA at a significance level of  $\alpha \leq 0.05$  showed that the treatment of encapsulating material ratios (alginate and photo-oxidized starch) and the ratio of encapsulating material to shallots in the production of shallot capsules had a significant effect on sample solubility (Figure 8).

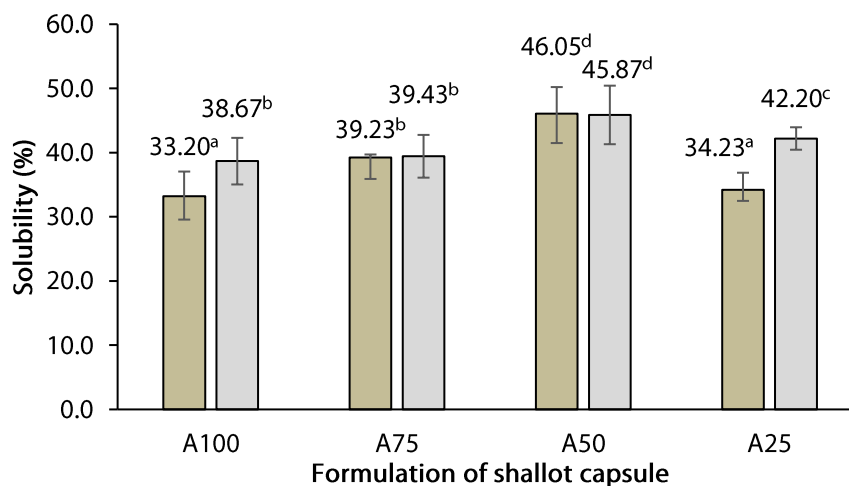


Figure 8. Solubility of shallot capsules from different ratios of shallot to encapsulant (■ 1:2 and □ 1:3)

Based on the results, the less alginate added, the higher the sample solubility. The sample with the highest solubility was A50K3 (46.05%), while the lowest was A100K3 (33.2%). This is due to the lower solubility of alginate compared to the solubility of photo-oxidized starch, which is 85.73% (Palupi et al., 2020). However, the lower solubility observed in the A25 compared to the A50 may be attributed to insufficient structural stability caused by the reduced alginate content. Alginate provides a gel matrix that supports encapsulation, and a significant reduction in its concentration could weaken the capsules, making them less capable of retaining their structure during solubility testing. This aligns with the findings of Yang et al. (2022), who reported that higher alginate concentrations increase cross-linking interactions with calcium ions, thereby reinforcing the gel structure. Conversely, reduced alginate content weakens the gel matrix, leading to lower solubility.

### **Triangle difference test**

A triangle difference test was conducted by 30 panelists on 2 different samples: A50K3 and fried shallots. The A50K3 sample was selected based on its high polyphenol content, and the fried shallot sample was chosen to compare the aroma of the shallot capsules with commercial fried shallots. A triangle test is useful for detecting differences between two products and can assess market trends, as well as the effects of changes in ingredients, packaging, processing methods, handling, or storage conditions (Marques et al., 2022). The results of the triangle difference test are shown in Table 2.

**Table 2.** Triangle difference test results for the aroma of shallot capsules and fried shallots

Response	Number of responses	Probability 0.1%
Correct	19	19
Incorrect	11	

Based on the results of the binomial one-tailed test, it was concluded that the shallot capsule and fried shallot samples were significantly different at a 0.1% significance level. Nineteen out of thirty panelists were able to distinguish the aroma of the shallot capsules from the fried shallots. The aroma of the shallot capsules was reduced due to the heating process and the addition of other materials during capsule preparation. According to Tantalulu et al. (2020), the flavor of shallots changes during processing. A study by Permatasari et al. (2022) found that storage temperature significantly affects the levels of Volatile Reducing Substances (VRS) in shallot powder, with higher temperatures accelerating the decrease in these compounds.

### **Conclusion**

Based on the research results, shallot capsules with a 1:3 ratio formulation showed better results compared to the results of the 1:2 ratio formulation. The 1:3 ratio formulation produced higher yields, polyphenols, and encapsulation efficiency compared to the 1:2 ratio formulation. The ratio of encapsulation materials used for the production of shallot capsules produced varying qualities. Samples with a 50:50 ratio produced the highest polyphenol content, encapsulation efficiency, load capacity, and solubility. The water content of the A50K2 treatment was the lowest, which was 0.790%. The formulation that produced the best quality shallot capsules was the A50K3 treatment, which contained 3.608 mg GAE/g of polyphenols and an encapsulation efficiency of 45.654%, the highest among the samples.

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