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MICROWAVE-ASSISTED RECOVERY AND SPRAY-DRYING ENCAPSULATION OF ASTAXANTHIN FROM SHRIMP SHELL BY-PRODUCTS: PHYSICOCHEMICAL PROPERTIES AND STORAGE STABILITY

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ABSTRACT

Astaxanthin is a high-value carotenoid with potent antioxidant activity; however, its recovery and stability remain major challenges due to its susceptibility to oxidation and thermal degradation. This study investigated the extraction of astaxanthin from shrimp shell by-products using microwave-assisted extraction (MAE) with soybean oil as a green solvent, followed by microencapsulation through spray drying to improve its stability. The effects of solid-to-oil ratio, microwave power, and irradiation time on astaxanthin recovery were evaluated. Astaxanthin yield increased with increasing extraction intensity and reached 33.78 µg/g at a solid-to-oil ratio of 1:4, microwave power of 30%, and irradiation time of 120 s. The astaxanthin-rich oil was subsequently encapsulated using a gum arabic–maltodextrin wall system. Encapsulation efficiency was significantly influenced by the core-to-wall ratio, inlet temperature, and feed rate, with the highest value of 74.26% obtained at a core-to-wall ratio of 1:4, an inlet temperature of 160 °C, and a feed rate of 1000 mL/h. The resulting powder contained 1.42 µg/g astaxanthin, exhibited low moisture content (3.12%), and showed good solubility (64.0%). Storage studies revealed that encapsulation effectively enhanced astaxanthin stability, with 82.5% of the initial pigment retained after 60 days at 25 °C, compared with 57.1% at 40 °C. The decrease in astaxanthin content was accompanied by changes in color parameters, indicating a close relationship between pigment degradation and color deterioration during storage. These findings demonstrate that soybean oil-assisted MAE coupled with spray-drying microencapsulation is an effective and environmentally friendly strategy for recovering and stabilizing astaxanthin from shrimp shell by-products. The developed process contributes to the valorization of shrimp-processing waste and provides a promising source of natural astaxanthin for food and nutraceutical applications.

Keywords: Astaxanthin, shrimp shell by-products, microwave-assisted extraction, spray drying, microencapsulation, storage stability.

1. INTRODUCTION

Shrimp processing generates substantial quantities of by-products, including heads, shells, and tails, which account for approximately 40 – 60% of the total biomass. These residues are frequently discarded or utilized as low – value animal feed despite being rich sources of proteins, minerals, chitin, and bioactive compounds [1]. As one of the world's leading shrimp-producing countries, Viet Nam produces large amounts of shrimp-processing waste annually, creating both environmental challenges and opportunities for value-added utilization. Among the bioactive compounds present in shrimp shells,

astaxanthin has attracted considerable attention because of its exceptional antioxidant activity and its potential applications in food, nutraceutical, pharmaceutical, and cosmetic products [2, 3].

The efficient recovery of astaxanthin remains a critical step for its industrial utilization. Conventional extraction methods typically employ organic solvents such as acetone, hexane, and ethanol. Although these solvents can achieve relatively high extraction yields, concerns regarding solvent residues, environmental impact, and additional solvent – removal steps have stimulated interest in greener extraction technologies. Microwave – assisted extraction (MAE) has emerged as a promising alternative because microwave energy generates rapid volumetric heating, enhances cell disruption, and accelerates mass transfer, thereby improving extraction efficiency while reducing extraction time and energy consumption [4, 5]. Among them, soybean oil is particularly attractive because it is food – grade, inexpensive, widely available, and exhibits a high solubilization capacity for hydrophobic carotenoids. Unlike conventional organic solvents, soybean oil can remain in the final formulation after extraction, thereby eliminating solvent – removal steps and reducing environmental concerns associated with solvent disposal. Furthermore, its direct compatibility with food systems facilitates the development of functional ingredients and nutraceutical formulations. Recent studies have also demonstrated the suitability of soybean oil as a green extraction medium for carotenoids and other lipophilic bioactive compounds [6]. Therefore, soybean oil was selected in the present study as a safe and sustainable extraction medium for astaxanthin recovery from shrimp shell by-products.

Despite its high biological value, astaxanthin is highly susceptible to oxidation, light exposure, and thermal degradation due to its highly unsaturated polyene structure. These stability issues limit its practical application and reduce its shelf life during processing and storage. Consequently, effective stabilization strategies are required to preserve its bioactivity and functionality. Among the available techniques, microencapsulation has been widely recognized as an effective approach for protecting sensitive bioactive compounds. Spray drying is particularly attractive because of its simplicity, scalability, continuous operation, and relatively low production cost [7].

The performance of spray – dried microcapsules is strongly influenced by the selection of wall materials. Gum arabic possesses excellent emulsifying and film-forming properties that facilitate the stabilization of oil droplets before drying. Maltodextrin, on the other hand, exhibits low viscosity, high water solubility, and good oxidative barrier properties. However, each material has certain limitations when used individually. Therefore, combinations of gum arabic and maltodextrin are frequently employed to exploit their complementary functionalities, resulting in improved emulsion stability, encapsulation efficiency, and storage stability of carotenoids and other lipophilic compounds [8]. Although numerous studies have investigated astaxanthin extraction and encapsulation separately, most extraction processes still rely on organic solvents, which limit direct food applications and require additional downstream processing. Furthermore, information regarding the use of soybean oil as a food-grade extraction medium in combination with microwave-assisted extraction remains limited. Studies integrating microwave – assisted oil extraction with subsequent spray – drying microencapsulation are also scarce, particularly for the recovery and stabilization of astaxanthin from shrimp shell by-products. Developing an integrated process that combines efficient extraction with effective stabilization could enhance astaxanthin recovery while improving its applicability in food systems.

Therefore, this study aimed to develop an integrated approach for the recovery and stabilization of astaxanthin from shrimp shell by – products. Microwave – assisted extraction using soybean oil was optimized to maximize astaxanthin recovery, and the resulting extract was subsequently microencapsulated by spray drying using a gum arabic – maltodextrin carrier system. The effects of spray – drying conditions on encapsulation efficiency and powder characteristics were also evaluated to assess the feasibility of producing a stable astaxanthin – rich powder suitable for food applications.

2. MATERIALS AND METHODS

2.1. Materials and chemicals

Shrimp shell by – products (*Litopenaeus vannamei*) were supplied by Minh Phu Seafood Corporation (Ca Mau Province, Viet Nam). Upon receipt, the shells were thoroughly washed with potable water to remove residual muscle tissues and adhering impurities. The cleaned shells were drained and dried in a forced-air oven at 50 °C until a constant weight was achieved. The dried material

was subsequently milled using a laboratory grinder and sieved through a 60 – mesh screen. The resulting shrimp shell powder was packed in polyethylene bags, protected from light, and stored at 4 °C until extraction.

Gum arabic (food grade) and maltodextrin (DE 10 – 12, food grade) used as encapsulating agents were purchased from Shandong Fuyang Biotechnology Co., Ltd. (Shandong, China). Astaxanthin standard (≥ 98% purity) was obtained from Sigma-Aldrich (St. Louis, MO, USA). Ethanol (HPLC grade, ≥ 99.9%) and methanol (HPLC grade) were purchased from Merck KGaA (Darmstadt, Germany). All other chemicals and reagents used in the study were of analytical grade.

2.2. Microwave-assisted extraction of astaxanthin

Astaxanthin was extracted from shrimp shell powder using a microwave-assisted extraction (MAE) method with slight modifications to the procedure described by Sachindra and Mahendrakar [9]. Briefly, shrimp shell powder was mixed with soybean oil at different solid-to-oil ratios (1:2, 1:3, 1:4, and 1:5, g/mL) in glass extraction vessels.

The extraction parameters were evaluated sequentially using a one-factor-at-a-time (OFAT) approach to investigate the individual effects of extraction variables on astaxanthin recovery and to identify suitable operating conditions for subsequent microencapsulation experiments. The effect of solid-to-oil ratio was first investigated while the remaining extraction conditions were kept constant. The ratio yielding the highest astaxanthin recovery was then selected for the evaluation of microwave power (0, 20, 30, and 40%). Subsequently, the microwave power associated with the highest recovery was used to examine the effect of microwave irradiation time (30, 60, 120, and 150 s).

Microwave treatments were carried out using a West Bend 3 – in – 1 Microwave Air Fryer Convection Oven operating at a frequency of 2.45 GHz with a rated microwave power output of 1000 W. The investigated power settings of 20%, 30%, and 40% corresponded to nominal power levels of approximately 200, 300, and 400 W, respectively. Following extraction, the mixtures were centrifuged at 5,000 rpm for 10 min (Hermle Z326K, Hermle Labortechnik GmbH, Germany). The astaxanthin-rich oil phase was collected, transferred into amber glass bottles, and stored at 4 °C until further analysis. The extraction condition producing the highest astaxanthin recovery was subsequently used for the preparation of spray-drying feed emulsions.

2.3. Conventional solvent extraction of astaxanthin

For comparison purposes, astaxanthin was extracted from shrimp shell powder using a conventional solvent extraction method adapted from Dahmoune et al., 2015 [10]. Ethanol 50% was selected as the reference solvent because aqueous ethanol systems have been widely employed for the extraction of carotenoids from biological matrices and represent a commonly used conventional extraction medium. In addition, the use of 50% ethanol was adopted from the reference method to provide a practical benchmark for evaluating the efficiency of the proposed microwave-assisted oil extraction process. Briefly, shrimp shell powder was mixed with ethanol 50% at a solid-to-solvent ratio of 1:10 (w/v) and extracted in a water bath at 60 °C for 2 hours under continuous stirring. After extraction, the mixture was centrifuged at 5,000 rpm for 10 min (Hermle Z326K, Hermle Labortechnik GmbH, Germany), and the supernatant was collected. The astaxanthin-containing extract was stored in amber glass bottles at 4 °C until further analysis. Astaxanthin content was determined according to the procedure described in Section 2.4. The astaxanthin recovery obtained by this conventional extraction method was 17.56 ± 0.2 μg/g dry shrimp shell powder.

2.4. Determination of astaxanthin content

Astaxanthin content was determined according to the method reported by Sachindra and Mahendrakar with minor modifications [9]. An aliquot of the extract was appropriately diluted with ethanol, and absorbance was measured at 474 nm using a UV – Visible spectrophotometer (UV – 1800, Shimadzu Corporation, Kyoto, Japan). Quantification was performed using a calibration curve prepared from standard astaxanthin solutions. Results were expressed as micrograms of astaxanthin per gram of dry shrimp shell powder (μg/g). Because absorbance was measured at a single wavelength (474 nm), the possible contribution of other carotenoids cannot be completely excluded.

2.5. Preparation of feed emulsion

The wall – material solution was prepared by dissolving gum arabic and maltodextrin in distilled water under continuous magnetic stirring at room temperature for 2 h. The solution was subsequently hydrated overnight at 4 °C to ensure complete dissolution and stabilization.

The astaxanthin extract obtained under the optimal extraction condition was incorporated into the wall – material solution. Emulsions were homogenized using a high-speed homogenizer (T25 Digital Ultra – Turrax, IKA – Werke GmbH & Co. KG, Staufen, Germany) at 10,000 rpm for 5 min. Different core – to – wall ratios (1:2, 1:3, and 1:4, w/w) were investigated to determine the optimal formulation for spray drying.

2.6. Spray drying

Microencapsulation of astaxanthin was performed using a laboratory – scale spray dryer (Mini Spray Dryer B-290, Büchi Labortechnik AG, Flawil, Switzerland). The influence of inlet air temperature (150, 160, and 170 °C) and feed rate (800, 1000, and 1200 mL/h) on encapsulation efficiency was evaluated. The atomized droplets were rapidly dried and collected through a cyclone separator. The resulting powders were immediately sealed in laminated aluminum pouches, protected from light, and stored in a desiccator at room temperature until analysis.

2.7. Determination of encapsulation efficiency

Encapsulation efficiency (EE) was determined according to the method of Akhavan Mahdavi et al. with slight modifications [11]. Surface astaxanthin was extracted by washing the powder with petroleum ether under gentle stirring. Total astaxanthin was determined after complete disruption of the microcapsules and extraction of the encapsulated pigment.

Encapsulation efficiency was calculated according to Equation (1):

$$EE\ (\%) = \frac{TA-SA}{TA} \times 100 \quad (1)$$

where TA is the total astaxanthin content and SA is the surface astaxanthin content.

2.8. Determination of powder yield

Powder yield (PY) was calculated as the ratio of the mass of powder collected after spray drying to the total solid content of the feed emulsion prior to drying. Powder yield was calculated according to Equation (2):

$$PY\ (\%) = (M_p / M_s) \times 100 \quad (2)$$

where M_p is the mass of powder collected after spray drying (g) and Ms is the total mass of solids present in the feed emulsion before drying (g).

2.9. Characterization of spray-dried powder

2.9.1. Moisture content

Moisture content was determined according to the method of Cano-Chauca et al. [12], based on AOAC Official Method 925.10. Approximately 2 g of powder was dried in a hot – air oven at 105 ºC until constant weight. Moisture content was calculated from the weight loss during drying and expressed as a percentage of the initial sample weight.

2.9.2. Solubility

Powder solubility was determined according to the method of Cano-Chauca et al. [12]. One gram of powder was dispersed in 100 mL of distilled water and stirred for 5 min. The suspension was centrifuged at 3000 rpm for 10 min. An aliquot of the supernatant was dried to constant weight, and solubility was calculated as the percentage of dissolved solids relative to the initial sample weight.

2.9.3. Water activity

Water activity (a_w) was determined using a water activity meter (AquaLab Series 4TE, Meter Group Inc., Pullman, WA, USA) at 25 ± 1 °C. Approximately 2 g of powder was placed in the sample cup and equilibrated in the measuring chamber until a stable reading was obtained.

2.9.4. Astaxanthin content in spray-dried powder

Astaxanthin content in the spray-dried powder was determined according to the method described in Section 2.4 with slight modifications. Briefly, 0.5 g of powder was dispersed in ethanol and stirred until complete dissolution of the wall materials. The resulting solution was filtered and appropriately diluted with ethanol prior to analysis. Absorbance was measured at 474 nm using a UV–Visible spectrophotometer (UV – 1800, Shimadzu Corporation, Kyoto, Japan). Astaxanthin concentration was quantified using a calibration curve prepared from standard astaxanthin solutions. Results were expressed as micrograms of astaxanthin per gram of powder (μg/g powder).

2.9.5. Color measurement

The color of the spray-dried astaxanthin powder was determined using a colorimeter (CR – 400, Konica Minolta, Tokyo, Japan). Color parameters were recorded according to the CIE L*a*b* color system, where L* represents lightness (0 = black, 100 = white), a* represents redness (+a*) to greenness (−a*), and b* represents yellowness (+b*) to blueness (−b*). Prior to measurement, the instrument was calibrated using a standard white calibration plate. Powder samples were placed in a sample holder to obtain a uniform surface, and measurements were performed at three different positions. Results were expressed as the mean values of L*, a*, and b*.

2.9.6. Fourier-transform infrared spectroscopy (FTIR)

The chemical characteristics of the spray-dried astaxanthin powder were analyzed using Fourier-transform infrared spectroscopy (FTIR) (Nicolet iS10, Thermo Fisher Scientific, Waltham, MA, USA). Powder samples were mixed with spectroscopic-grade potassium bromide (KBr) and compressed into pellets prior to analysis. FTIR spectra were recorded over the range of 4000 – 400 cm^{-1} at a resolution of 4 cm^{-1} with 32 scans per sample. The obtained spectra were used to identify the major functional groups present in the spray – dried powder and to evaluate possible interactions between the wall materials and the encapsulated astaxanthin – rich oil.

2.9.7. Storage stability

The spray – dried astaxanthin powder was packaged in laminated aluminum pouches and stored at 25 ± 2 °C or 40 ± 2 °C under controlled relative humidity conditions (55 ± 2% RH) for 60 days. Samples were withdrawn at 15 – day intervals to determine astaxanthin retention and color parameters (L*, a*, and b*). All samples were protected from direct light during storage.

2.10. Statistical analysis

All experiments were performed in triplicate, and results were expressed as mean ± standard deviation. Statistical analyses were conducted using SPSS software version 22.0 (IBM Corp., Armonk, NY, USA). Significant differences among treatments were determined by one-way analysis of variance (ANOVA) followed by Duncan's multiple range test at a significance level of $p < 0.05$.

3. RESULTS AND DISCUSSION

3.1. Effect of extraction parameters on astaxanthin recovery

The extraction parameters significantly influenced astaxanthin recovery from shrimp shell by-products (Table 1). Astaxanthin recovery obtained by microwave-assisted extraction (MAE) ranged from 18.61 to 33.78 μg/g depending on the extraction conditions and exceeded that obtained by conventional solvent extraction using 50% ethanol (17.56 ± 0.2 μg/g) under most conditions. The higher recovery achieved by MAE can be attributed to the combined effects of microwave-induced disruption of the shrimp shell matrix and the ability of soybean oil to effectively dissolve lipophilic carotenoids. In addition to improving extraction efficiency, soybean oil provides a food-grade extraction medium that can be directly used in subsequent processing without solvent removal.

Table 1. Effect of microwave-assisted extraction parameters on astaxanthin recovery from shrimp shell by-products

Parameter	Value	Astaxanthin (μg/g)
Solid-to-oil ratio (g/mL)	1:2	18.61 ± 0.3^{c}
	1:3	24.55 ± 0.4^{b}
	1:4	31.82± 0.4^{a}
	1:5	31.03± 0.5^{a}
Microwave power (%)	0	3.44± 0.1^{d}
	20	28.66± 0.5^{c}
	30	33.12± 0.4^{a}
	40	30.77± 0.5^{b}
Microwave irradiation time (s)	30	24.74± 0.4^{d}
	60	28.93± 0.3^{c}
	120	33.78± 0.1^{a}
	150	32.14± 0.4^{b}

Different superscript letters within the same parameter indicate significant differences ($p < 0.05$).

The solid-to-oil ratio significantly affected astaxanthin recovery. Increasing the ratio from 1:2 to 1:3 increased astaxanthin content from 18.61 to 24.55 μg/g, while a further increase to 1:4 resulted in the highest recovery of 31.82 μg/g. Compared with the 1:2 treatment, this corresponded to an increase of approximately 71%. The greater availability of extraction medium enhanced the dissolution and diffusion of astaxanthin released from the shrimp shell matrix into the oil phase. Because astaxanthin is highly hydrophobic, an adequate volume of oil is required to maintain a concentration gradient that promotes mass transfer from the solid matrix into the extraction medium. Similar behavior has been reported during carotenoid extraction from crustacean by-products, where increasing solvent volume improved extraction efficiency by facilitating pigment solubilization and diffusion [3]. Notably, even the lowest MAE treatment (18.61 μg/g) slightly exceeded the recovery obtained by conventional solvent extraction (17.56 μg/g), despite the much shorter extraction time. However, increasing the ratio to 1:5 did not significantly improve astaxanthin recovery, yielding 31.03 μg/g. The absence of a significant increase beyond the 1:4 ratio indicates that the extraction system had approached equilibrium, and additional oil contributed little to further carotenoid recovery while increasing processing costs.

Microwave power exerted a pronounced influence on astaxanthin recovery. In the absence of microwave irradiation, only 3.4 μg/g astaxanthin was recovered, indicating limited diffusion of the pigment into the oil phase under passive extraction conditions. Increasing microwave power from 20 to 30% significantly enhanced recovery from 28.66 to 33.12 μg/g. At 30% power, astaxanthin recovery was approximately 9.7 times higher than that of the untreated control and nearly 88% higher than that obtained by conventional solvent extraction. The increase in recovery is consistent with the rapid volumetric heating generated by microwave irradiation, which promotes internal pressure buildup within biological tissues, disrupts cellular structures, and accelerates mass transfer of intracellular compounds into the surrounding medium [13]. In shrimp shell tissues, microwave energy may weaken interactions between astaxanthin and structural components such as proteins and chitin, thereby facilitating pigment release. Recent studies have further suggested that microwave treatment enhances solvent penetration and accelerates the diffusion of lipophilic compounds through localized heating and pressure gradients within biological matrices, thereby improving extraction efficiency while substantially reducing processing time [10]. Similar enhancements in carotenoid extraction under moderate microwave conditions have been reported for marine by-products and plant materials. However, increasing microwave power to 40% resulted in a slight decline in recovery to 30.77 μg/g. This reduction may be associated with thermal degradation and oxidation of astaxanthin caused by excessive energy input [10]. Due to its highly unsaturated polyene structure, astaxanthin is particularly susceptible to oxidation and trans – cis isomerization under elevated temperatures, leading to partial degradation of the pigment [3].

A similar trend was observed for microwave irradiation time. Extending the irradiation period from 30 to 60 s increased astaxanthin recovery from 24.7 to 28.9 μg/g, while further extension to 120 s produced the highest recovery of 33.78 μg/g. Longer irradiation times promoted more extensive disruption of the shrimp shell matrix and improved heat transfer throughout the sample, thereby facilitating the release and diffusion of astaxanthin into the oil phase. Compared with conventional solvent extraction, the recovery obtained after only 120 s of microwave treatment was approximately 92% higher, despite reducing extraction time from 2 h to 2 min. The marked increase in recovery obtained within such a short processing time indicates that microwave energy substantially accelerates extraction kinetics relative to conventional heating. However, prolonging irradiation to 150 s did not significantly improve recovery, and astaxanthin content decreased slightly to 32.14 μg/g. The reduction suggests that prolonged exposure to microwave energy may accelerate degradation reactions that partially offset the benefits of increased extraction intensity [9, 10]. Similar decreases in carotenoid recovery at excessive extraction times have been reported previously and are generally attributed to thermal and oxidative degradation of carotenoid molecules [10]. It should be noted that astaxanthin content was determined by UV – Vis spectrophotometry at 474 nm using an astaxanthin standard calibration curve. Although minor interference from other carotenoids cannot be completely excluded, the use of an astaxanthin standard calibration curve and identical analytical conditions for all treatments provides a reliable basis for comparative evaluation of extraction performance.

Based on the findings, a solid-to-oil ratio of 1:4, microwave power of 30%, and irradiation time of 120 s yielded the highest astaxanthin recovery (33.78 μg/g) and were therefore selected for subsequent microencapsulation experiments. Under these conditions, astaxanthin recovery was approximately 92% higher than that obtained by conventional solvent extraction (17.56 μg/g), while reducing extraction time from 2 h to only 120 s.

3.2. Effect of spray-drying parameters on encapsulation efficiency

The encapsulation efficiency (EE) and powder yield (PY) of astaxanthin microcapsules were significantly affected by spray-drying conditions, ranging from 38.71–74.26% and 70.91–78.83%, respectively (Table 2). These results indicate that spray-drying parameters influenced not only astaxanthin retention but also powder recovery during the drying process. The SEM observations (Figure 1) further confirmed that changes in processing conditions altered particle morphology, which in turn influenced the effectiveness of astaxanthin entrapment within the wall matrix.

Among the investigated factors, the core-to-wall ratio markedly influenced encapsulation performance. Increasing the ratio from 1:2 to 1:3 increased EE from 45.40% to 68.11%, corresponding to an improvement of approximately 50%, while a further increase to 1:4 resulted in the highest EE of 73.14%. Compared with the 1:2 formulation, the EE of the 1:4 treatment increased by more than 61%, demonstrating the importance of providing sufficient wall material for effective astaxanthin entrapment. Powder yield (PY) was also significantly affected by the core-to-wall ratio, increasing from 74.54% at 1:2 to 78.24% at 1:3 before slightly decreasing to 76.13% at 1:4. The higher PY observed at intermediate wall-material levels may be attributed to improved particle formation and more efficient powder collection during spray drying.

Table 2. Effect of spray-drying parameters on the encapsulation efficiency of astaxanthin powder

Parameter	Value	PY (%)	EE (%)
Core-to-wall ratio	1:2	74.54 ± 0.82^{c}	45.40 ± 0.74^{c}
	1:3	78.24 ± 0.82^{a}	68.11 ± 0.42^{b}
	1:4	76.13 ± 0.62^{b}	73.14 ± 0.72^{a}
Inlet temperature (°C)	150	71.52 ± 0.54^{c}	51.0 ± 0.8^{c}
	160	78.83 ± 0.51^{a}	73.18 ± 0.65^{a}
	170	75.15 ± 0.42^{b}	66.5 ± 0.7^{b}
Feed rate (mL/h)	800	70.91 ± 0.86^{b}	58.2 ± 0.7^{b}
	1000	77.95 ± 0.85^{a}	74.26 ± 0.55^{a}
	1200	76.93 ± 1.15^{a}	38.71 ± 0.94^{c}

Different superscript letters within the same parameter indicate significant differences ($p < 0.05$).

The lower EE obtained at the 1:2 ratio suggests that the available gum arabic–maltodextrin matrix was insufficient to completely cover the dispersed astaxanthin droplets generated during atomization, thereby increasing the proportion of astaxanthin located near the particle surface and susceptible to oxidation during drying. SEM observations supported this interpretation, as particles produced at lower wall-material levels exhibited less uniform surfaces and a greater incidence of surface irregularities (Figure 1.A1). In contrast, increasing the wall-material proportion resulted in particles with smoother surfaces and fewer visible defects, particularly at the 1:4 ratio (Figure 1.A3). The improved surface integrity suggests the formation of a more continuous encapsulating matrix, which likely reduced astaxanthin diffusion toward the particle surface and enhanced its retention during drying. Similar effects of increasing wall-material concentration have been reported for spray-dried carotenoid and lipid-based bioactive compounds, where thicker matrix formation resulted in improved encapsulation efficiency [14, 15].

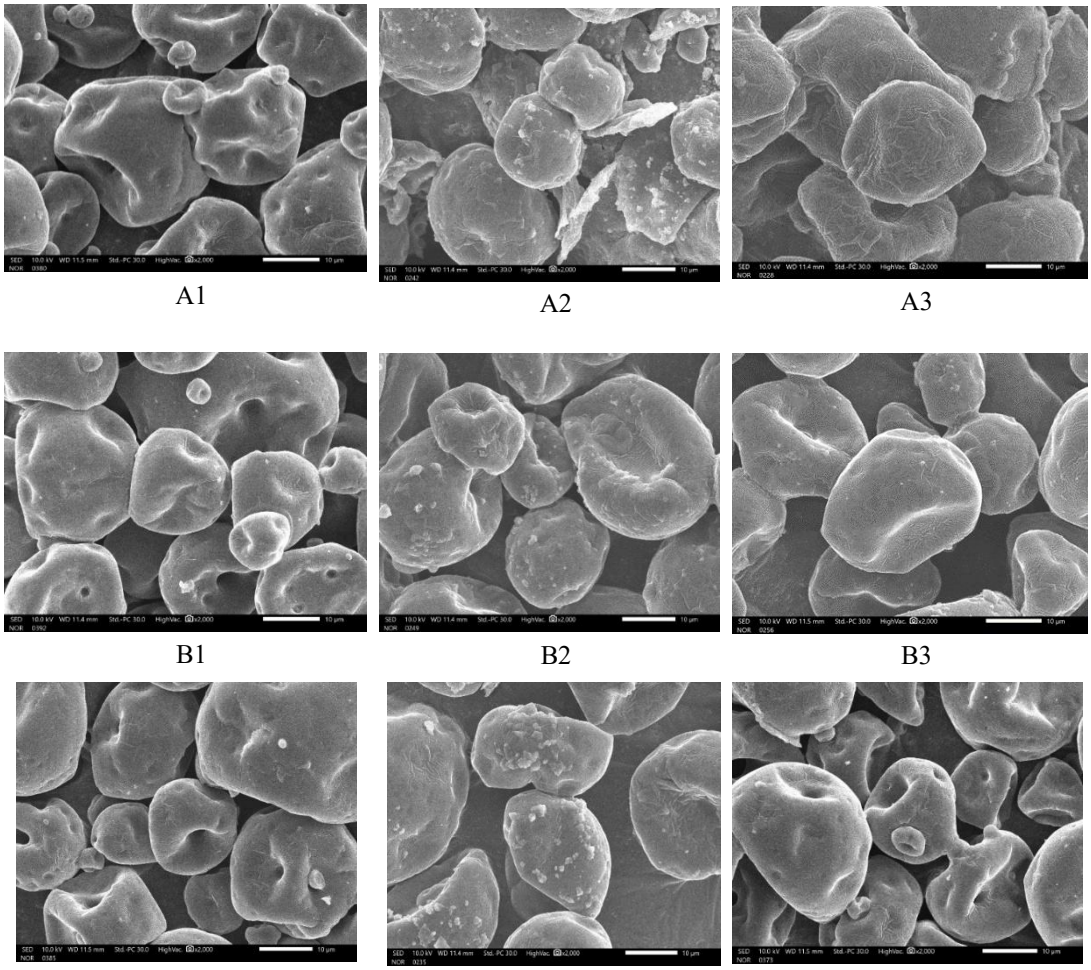

Figure 1. SEM images of spray-dried astaxanthin microcapsules prepared under different core – to – wall ratios (A1 – A3), inlet air temperatures (B1 – B3), and feed rates (C1 – C3)

Inlet temperature also significantly affected EE. Raising the drying temperature from 150 to 160 °C increased EE from 51.0% to 73.18%, representing an increase of approximately 43.5%. However, a further increase to 170 °C reduced EE to 66.5%. A similar trend was observed for PY, which increased from 71.52% at 150 °C to 78.83% at 160 °C and subsequently decreased to 75.15% at 170 °C. The higher PY at 160 °C suggests more efficient moisture removal and particle collection, whereas excessive drying temperatures may increase particle losses due to wall deposition and structural damage. These results indicate that 160 °C provided the most favorable balance between rapid moisture removal and

astaxanthin stability. At 150 °C, the relatively low EE may be attributed to slower evaporation rates, which prolonged the period during which dissolved astaxanthin could migrate toward the droplet surface before complete crust formation. Consistent with this explanation, particles obtained at 150 °C exhibited noticeable surface shrinkage and structural deformation (Figure 1.B1), indicating insufficient shell development during drying. Increasing the temperature to 160 °C promoted rapid crust formation and generated particles with smoother and more intact surfaces (Figure 1.B2), contributing to improved astaxanthin entrapment. However, further increasing the temperature to 170 °C led to the appearance of more pronounced surface indentations and collapse (Figure 1.B3), suggesting increased internal stress associated with excessive moisture evaporation. Such structural defects may facilitate the migration of encapsulated astaxanthin toward the particle surface and partially explain the reduction in EE. Furthermore, astaxanthin is highly susceptible to thermal oxidation because of its extensive conjugated double-bond system, which may further contribute to the decline in EE at elevated temperatures [2, 3].

Among all investigated variables, feed rate produced the largest variation in encapsulation efficiency, indicating that drying kinetics exerted a stronger influence on astaxanthin retention than either wall – material concentration or inlet temperature. Increasing the feed rate from 800 to 1000 mL/h enhanced EE from 58.2% to 74.26%, whereas a further increase to 1200 mL/h caused a dramatic reduction to only 38.71%. Compared with the optimal condition, the EE at 1200 mL/h decreased by nearly 48%, highlighting the sensitivity of particle formation to drying capacity. Feed rate also affected PY, which increased from 70.91% at 800 mL/h to 77.95% at 1000 mL/h and remained relatively unchanged at 1200 mL/h (76.93%). Although powder collection efficiency remained high at 1200 mL/h, the substantial reduction in EE indicates that rapid feed delivery adversely affected astaxanthin retention to a much greater extent than powder recovery. The superior EE obtained at 1000 mL/h suggests that this condition provided an optimal balance between liquid feed input and the evaporation capacity of the system, allowing sufficient moisture removal and effective shell formation. SEM observations clearly supported this trend. Particles produced at 1000 mL/h displayed relatively smooth surfaces and a more intact structure (Figure 1.C2), whereas particles obtained at 800 mL/h showed greater surface shrinkage, likely due to prolonged exposure to drying air (Figure 1.C1). In contrast, particles produced at 1200 mL/h exhibited obvious deformation and surface collapse (Figure 1.C3), indicating incomplete drying and insufficient structural stabilization before collection. These morphological defects can increase the exposure of astaxanthin at or near the particle surface, thereby promoting oxidation and reducing encapsulation efficiency. Excessive feed rates increase droplet loading within the drying chamber and shorten the effective drying time available for moisture removal, resulting in particles with higher residual moisture and weaker wall structures [16].

Based on these findings, both EE and PY were closely associated with particle morphology observed by SEM. Treatments producing smoother, more spherical, and structurally intact microcapsules consistently exhibited higher encapsulation efficiencies and improved powder recovery. Among the investigated conditions, a core-to-wall ratio of 1:4, an inlet temperature of 160 °C, and a feed rate of 1000 mL/h generated the most favorable particle structure together with high EE and PY values, and were therefore selected for subsequent characterization and application studies. Under the extraction conditions yielding the highest astaxanthin recovery, astaxanthin content reached 33.78 μg/g shrimp shell powder. Subsequent spray drying using the selected encapsulation conditions produced a powder containing 1.42 μg astaxanthin/g with an encapsulation efficiency of 74.26% and a powder yield of 77.95%, demonstrating the successful incorporation of the extracted astaxanthin into the microcapsule matrix.

3.3. Physicochemical characteristics and storage stability of spray – dried astaxanthin powder

The physicochemical characteristics of the spray-dried astaxanthin powder are presented in Table 3. The powder contained 1.42 μg/g astaxanthin and exhibited an encapsulation efficiency of 74.26%, indicating that a substantial proportion of the extracted astaxanthin was retained during spray drying. The relatively low astaxanthin concentration on a powder basis can be attributed to the high proportion of wall materials used during microencapsulation, which diluted the active compound within the final powder matrix.

The powder exhibited a low moisture content (3.12%) and water activity (a_w = 0.28), indicating efficient water removal during spray drying and favorable conditions for storage stability. Moisture contents below 5% and low aw values are generally considered desirable for spray-dried powders because they limit molecular mobility, microbial growth, and degradation reactions associated with lipid

oxidation and pigment deterioration. The combination of low moisture content and low aw therefore suggests that the powder possessed suitable characteristics for extended storage. In addition, the powder exhibited a solubility of 64.0%, indicating its potential suitability for incorporation into aqueous food systems. Similar physicochemical properties have been reported for spray-dried carotenoid powders and are considered favorable for maintaining product quality during storage [15, 16].

Table 3. Characteristics of spray-dried astaxanthin powder

Parameter	Value
Astaxanthin content (μg/g)	1.42 ± 0.07
Encapsulation efficiency (%)	74.26 ± 0.55
Moisture content (%)	3.12 ± 0.08
Water activity (a_w)	0.28 ± 0.01
Solubility (%)	64.0 ± 2.8

The FTIR spectrum of the spray – dried astaxanthin powder is shown in Figure 2. A broad absorption band at approximately 3293 cm^{-1} was attributed to O–H stretching vibrations associated with hydroxyl groups of gum arabic and maltodextrin [17]. The absorption band at 2923 cm^{-1} corresponded to C–H stretching vibrations of aliphatic groups, while the peak at 1745 cm^{-1} was assigned to C=O stretching vibrations, which may be associated with ester groups originating from soybean oil and astaxanthin esters [17]. The band observed at 1649 cm^{-1} was related to bound water and hydrogen – bond interactions within the matrix [17]. In addition, the absorption bands at 1147 and 1015 cm^{-1} were characteristic of C–O–C and C–O stretching vibrations of polysaccharides [17]. The FTIR spectrum exhibited the characteristic functional groups of the wall materials and oil phase without the appearance of additional absorption bands that could indicate the formation of new covalent bonds. These observations provide indirect evidence that the astaxanthin – rich oil was successfully incorporated into the gum arabic – maltodextrin matrix and are consistent with encapsulation occurring primarily through physical entrapment during spray drying. However, FTIR analysis alone is not sufficient to conclusively confirm the encapsulation mechanism or molecular interactions between the core and wall materials. Additional analytical techniques, such as DSC, XRD, NMR, or microscopic analyses, would be required to further elucidate the encapsulation mechanism.

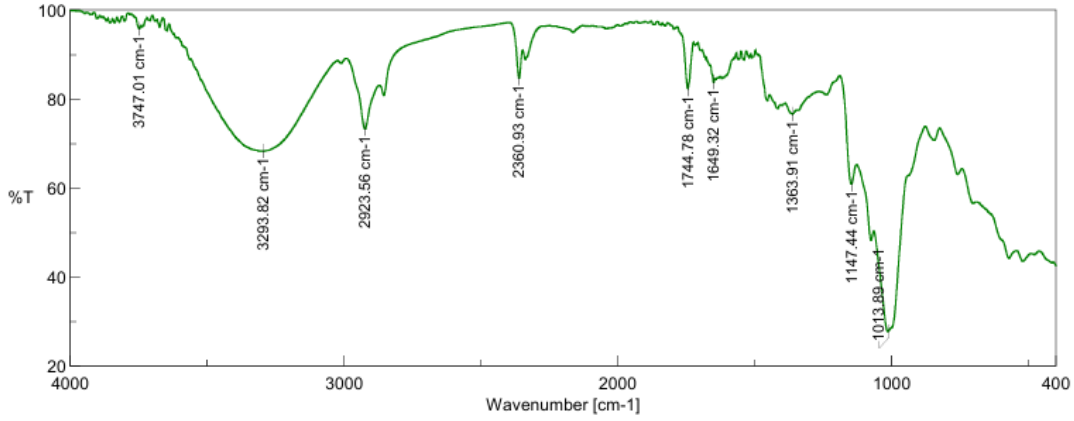

Figure 2. FTIR spectrum of spray-dried astaxanthin microcapsules prepared with gum arabic and maltodextrin

The stability of encapsulated astaxanthin was further evaluated during storage at 25 and 40 °C (Table 4). Astaxanthin retention gradually decreased with storage time under both conditions; however, the degradation rate was strongly influenced by storage temperature. At 25 °C, astaxanthin retention remained relatively high, decreasing from 100% to 82.5% after 60 days. In contrast, storage at 40 °C accelerated degradation, resulting in only 57.1% retention after the same period. The greater loss observed at elevated temperature is consistent with the known susceptibility of astaxanthin to oxidation,

isomerization, and thermal degradation due to its highly conjugated polyene structure [2], [3]. Although degradation occurred under both storage conditions, the relatively high retention observed at 25 °C suggests that the gum arabic–maltodextrin matrix provided effective protection against environmental factors, thereby slowing pigment deterioration.

Changes in astaxanthin retention were accompanied by significant variations in color parameters. The a* value, which reflects redness, progressively decreased during storage and closely followed the reduction in astaxanthin content. At 25 °C, a* decreased from 18.4 to 15.1 after 60 days, whereas a more pronounced decline to 11.4 was observed at 40 °C. The reduction in redness is characteristic of carotenoid degradation and reflects the gradual loss of the red – orange pigments responsible for the characteristic color of astaxanthin. Conversely, L* values increased throughout storage, indicating a lighter appearance of the powder as pigment concentration decreased. Similar relationships between carotenoid degradation and color deterioration have been reported in spray – dried powders containing astaxanthin, β – carotene, and other carotenoid – rich extracts [14]. Notably, the changes in color closely mirrored the degradation pattern of astaxanthin, suggesting that color measurements may provide a rapid and practical indicator of pigment stability during storage. The stronger decline in both astaxanthin retention and a* values at 40 °C further confirms the detrimental effect of elevated temperature on carotenoid preservation. These findings highlight the importance of controlling storage conditions and indicate that the gum arabic–maltodextrin matrix effectively protected astaxanthin during storage, particularly at 25 °C. Because storage was conducted under controlled relative humidity conditions (55% RH), the observed differences in astaxanthin retention primarily reflected the effect of temperature. However, the influence of other environmental factors, including oxygen exposure and light intensity, was not investigated and warrants further study.

Table 4. Changes in astaxanthin retention and color parameters of spray-dried astaxanthin powder during storage at different temperatures

Storage temperature (°C)	Time (days)	Astaxanthin retention (%)	L*	a*	b*
25	0	100.00 ± 0.00^{a}	68.2 ± 0.3	18.4 ± 0.1	21.4 ± 0.5
	15	95.6 ± 0.8^{b}	68.9 ± 0.5	17.7 ± 0.3	20.8 ± 0.3
	30	91.3 ± 1.1^{c}	69.8 ± 0.4	16.8 ± 0.2	20.1 ± 0.7
	45	86.7 ± 0.9^{d}	70.6 ± 0.1	15.9 ± 0.5	19.4 ± 0.4
	60	82.5 ± 1.1^{e}	71.2 ± 0.7	15.1 ± 0.4	18.5 ± 0.1
40	0	100.00 ± 0.00^{a}	68.2 ± 0.2	18.4 ± 0.3	21.4 ± 0.2
	15	89.2 ± 1.0^{b}	70.1 ± 0.4	16.5 ± 0.6	19.8 ± 0.3
	30	77.4 ± 1.3^{c}	71.9 ± 0.7	14.7 ± 0.8	18.2 ± 0.7
	45	66.8 ± 1.2^{d}	73.2 ± 0.5	13.0 ± 0.4	16.8 ± 0.4
	60	57.1 ± 1.5^{e}	74.5 ± 0.6	11.4 ± 0.2	15.3 ± 0.5

Different superscript letters within the same parameter indicate significant differences ($p < 0.05$).

4. CONCLUSION

Microwave-assisted extraction using soybean oil was shown to be an effective approach for recovering astaxanthin from shrimp shell by – products. Astaxanthin recovery was significantly influenced by extraction conditions and reached the highest value at a solid – to – oil ratio of 1:4, microwave power of 30%, and irradiation time of 120 s. Moderate microwave treatment enhanced the release of astaxanthin from the shrimp shell matrix, whereas excessive extraction intensity reduced astaxanthin recovery, likely due to pigment degradation. The extracted astaxanthin – rich oil was successfully incorporated into a gum arabic – maltodextrin carrier system and stabilized through spray – drying microencapsulation. Among the investigated conditions, a core – to – wall ratio of 1:4, an inlet temperature of 160 °C, and a feed rate of 1000 mL/h produced the highest encapsulation efficiency and powder yield. The resulting powder exhibited low moisture content, low water activity, good solubility, and satisfactory astaxanthin retention during storage. Storage stability was strongly affected by

temperature, with astaxanthin retention remaining higher at 25 °C than at 40 °C throughout the 60 – day storage period. Changes in color parameters closely followed astaxanthin degradation, indicating that color measurements may be useful as a rapid indicator of pigment stability during storage. This study showed the potential of soybean oil – assisted microwave extraction combined with spray – drying microencapsulation for the recovery and stabilization of astaxanthin from shrimp shell by – products. Although FTIR analysis provided indirect evidence of the incorporation of astaxanthin into the gum arabic – maltodextrin matrix, further studies using complementary analytical techniques are required to better understand the encapsulation mechanism and molecular interactions between the core and wall materials. Additional investigations on extraction yield, degradation kinetics, bioavailability, and performance in food systems are also needed before practical applications can be established.

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