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Effect of Wall Material Concentrations on Microencapsulation Efficiency and Oxidative Stability of Pomegranate Seed Oil

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ABSTRACT

The purpose of this study is to evaluate how different compositions of wall materials affect the efficiency and oxidative stability of pomegranate seed oil (PSO) by spray drying. Three different wall materials (maltodextrin, concentrated milk protein, and lecithin) at three ratios: 1:3 (A), 1:3.5 (B), and 1:4 (C) (oil: wall material) are taken into consideration. All emulsions showed stability over 24 h, with the highest viscosity ratio observed at the 1:4 ratio. The properties of the encapsulated powders were analyzed and the microencapsulation efficiency (MEE) was evaluated at 91.60%, the solubility was 87.66%, the moisture content 2.33 g/100 g, and the bulk density was 0.65 g/cm^3. The particle size did not exceed 100 μm. All the particles were spherical, proportionate in size, and free of cracks. A storage stability test, conducted on the encapsulated oil for 28 days with 7 days intervals, showed that the antioxidant content was 69.30% and the peroxide value was 4.00meq/kg compared to free oil, PV was 42.03 meq/kg and DPPH was 55.33%. However, the total phenolic content (TPC) showed a significant decrease in free oil compared to the encapsulated oil with values of 0.57 mg GAE/g and 0.90 mg GAE/g for the encapsulated oil.

1 | Introduction

Contemporary consumers consider increasingly care about their health and well-being leading to a growing craving for food enriched with functional ingredients that enhance both nutritional and health value and physiological benefits. There has been increased interest in vegetable oils due to their health and nutritional properties in food pharmaceutical and cosmetics sectors (Mohammed et al. 2020). The food industry utilizes pomegranate fruit in large quantities for the manufacture of juices and jams, flavorings, and beverage colorings pomegranate seeds are removed during processing and considered waste wasting their valuable content. However, studies indicate the possibility of considering these wastes as a rich source of biologically active compounds and nutrients that can be extracted and used, in line according to the European Union's closed-loop policy, sustainable development goals, and the concept of zero or less waste (Siol et al. 2024). Interest has grown in the sustainable utilization of fruit and vegetable waste due to its valuable bioactive compounds such as phenols, fatty acids, and dietary fiber, which possess antioxidant and health-beneficial properties, making them suitable for developing functional and healthy foods, therefore, utilizing pomegranate seed byproducts by extracting their oil and packaging it into flour is a sustainable strategy to transform these byproducts into high-value and stable food components (Panda et al. 2025).

Pomegranate seeds represent about (3.7–7.9 g per 100 g of fruit mass) and are a rich source of fat (12–20 g per 100 g) with a distinct chemical composition of compounds that are biologically active (Costa et al. 2020). Pomegranate seed oil (PSO) is considered one of the most prominent vegetable oils as it has many health benefits and contains antioxidants that contribute to reducing the incidence of diabetes, lowering blood pressure, reducing obesity, improving heart health, promoting skin health, relieving the symptoms of rheumatoid arthritis and others (Kori et al. 2022). It also has high levels of health-promoting unsaturated fatty acids. For example, punicic acid makes up between 65% and 85% of the oil's fatty acid content. The oil also contains a wide range of antioxidant compounds including tocopherols, polyphenols, and sterols, at levels that exceed those found in edible oils (Tavakoli et al. 2024). Pomegranate essential oil is considered one of the oils that are subject to oxidation during processing and storage processes which may lead to changes in physical properties such as color, texture, and other sensory qualities, in addition to chemical changes that affect nutritional value and molecular activity, and may ultimately produce harmful compounds such as hydroperoxides, aldehydes, ketones, and free radicals (Drinić et al. 2020). Therefore, an encapsulation technology is used to help improve the protocols and oxidation in addition to improving the surface appearance (Paul and Radhakrishnan 2020). This is done by protecting it from direct exposure to oxygen and oxidizing stimulants such as light moisture and heat (Nejatian et al. 2024). Microencapsulation is defined as the process of encapsulating a single active ingredient, known as the matrix, within a wall material, enhancing the oxidative stability and functional properties of the oil (Pattnaik and Mishra 2022). Spray drying technology is considered one of the oldest and most common microencapsulation methods in the food industry due to its ability to operate continuously and low production costs (Mohammed et al. 2020). The effectiveness of oil encapsulation using this technique depends largely on the selection of appropriate wall materials and the properties of the oil-in-water (O/W) emulsion used during drying. Carbohydrates such as Gum Arabic (GA) and maltodextrin (MD), in addition to certain proteins, are often used as wall materials in encapsulation processes. These materials, whether used individually or in mixtures are characterized by their ability to achieve high encapsulation efficiency and contribute to improving the stability of the encapsulated compounds (Bajac et al. 2022). Among these materials used in microencapsulation, maltodextrin, a molecular hydrolysis product of starch, is widely used as a wall material in food packaging, essential oils, and pharmaceuticals. It boasts low viscosity, high solubility, and low cost, as well as good thermal stability and the ability to form a solid film around oil droplets during drying. However, its limited emulsifying ability requires blending with other coating materials such as whey protein, Gum Arabic, or lecithin (Xiao et al. 2022). Concentrated milk protein and milk components are used in packaging materials as they have desirable properties that make the encapsulated components functional and effective. They have beneficial emulsifying properties to protect the active ingredients and form stable emulsions (Augustin et al. 2010). Lecithin is a lipophilic phospholipid containing both a hydrophilic and a lipophilic component making it effective in reducing the surface tension between oil and water, thereby improving emulsion stability. It stabilizes oil droplets in a complex water wall preventing them from agglomerating and coalescing before drying (Chaabane et al. 2022). The main objective of this study is to manufacture microcapsules to protect PSO from oxidation and to study its storage stability and physical and chemical properties.

This study presents a novel contribution in the field of PSO encapsulation by employing an unconventional ternary combination of wall materials (maltodextrin, concentrated milk protein, and lecithin) in specifically studied ratios (1:3 to 1:4). Unlike previous studies that commonly used only two materials or used different encapsulation materials, this work emphasizes the synergistic effect of the three-component formulation on emulsion stability, encapsulation efficiency, solubility, and storage stability. Furthermore, it is among the few studies to integrate ultrasonic oil extraction using ethanol with microencapsulation. A comprehensive evaluation of the resulting powder was conducted, focusing on particle morphology, size distribution, and oxidative stability over a 28 days storage period. This research supports the sustainable and effective exploitation of food industry waste to develop functional and nutritional delivery systems.

2 | Materials and Methods

2.1 | Materials

Pomegranate seeds were obtained from juice vendors in Tikrit, Iraq. Pure ethanol, obtained from the German company Huneol, was used as a solvent and the wall materials were maltodextrin (Qinhuangdao Lihua Starch, China), concentrated milk protein (SOLAGO, Ireland), and lecithin (Srighem, India).

2.2 | PSO Extraction

PSO was extracted using an ultrasonic-assisted extraction (UAE) method with absolute ethanol as a green solvent following the procedure described by Li et al. (2015) with slight modifications. Briefly, pomegranate seed powder was mixed with ethanol at a ratio of 1:4 (w/v) in a 1 L beaker and stirred continuously using a magnetic stirrer at 40°C for 40 min. The mixture was then ultrasonically treated using an ultrasonic device (Lab Tech, made in Korea) at a temperature not exceeding 40°C for 20 min. After ultrasonic treatment the mixture was filtered through a fine porcelain cloth and the sediment was washed off by adding an additional 15% of the solvent volume to ensure complete extraction. The collected filtrate was then centrifuged at 4032 × g at 25°C for 8 min (Model 2010, made in Japan) The solvent-removed filtrate was collected using a rotary evaporator at 45°C and low pressure. The resulting oil was then filtered through a 0.45 μm membrane filter and stored in a tightly sealed, opaque glass container in a refrigerator at 4°C.

The extraction yield (%) was calculated using the following equation:

$$\text{Yield of oil (\%)} = \frac{\text{mass of extracted oil}}{\text{mass of bran}} \times 100\%.$$

2.3 | Emulsion Preparation

The emulsion was prepared according to the method described by Mohammed et al. 2021. The emulsion was prepared at three different ratios 1:3 (A), 1:3.5 (B), and 1:4 (C) to determine the optimal concentration for the best emulsion stability. Table 1 shows the proportions of the wall materials. Initially, maltodextrin, and concentrated milk protein were dissolved in distilled water with continuous stirring using a magnetic stirrer to ensure complete dissolution and homogeneous distribution of the components. Stirring continued overnight (12 h) at room temperature to ensure complete dissolution of the wall materials. After the dissolution process was complete, a lecithin and oil mixture was prepared separately. Lecithin was mixed at a rate of 1% of the total wall material proportions, and this mixture was then gradually added to the maltodextrin and milk protein concentrate solution under continuous stirring to ensure homogeneous distribution of the oil in the aqueous phase and the formation of a stable primary emulsion. Finally, the resulting mixture was homogenized using a homogenizer at a speed of 8000 rpm for 10 min, which led to a reduction in the size of the oil droplets and their uniform distribution within the aqueous medium to form a homogeneous and stable emulsion ready for the spray drying process.

2.4 | Emulsion Characterization

2.4.1 | Emulsion Stability

After the emulsion was prepared, the stability of each sample emulsion was checked by placing 25 mL of the emulsion into 50 mL placed in graduated cylinders at room temperature for 24 h. The stability of the emulsions was evaluated by monitoring phase separation during the 24 h following homogenization (Mohammed, Meor Hussin, et al. 2017).

2.4.2 | Emulsion Viscosity

The value of the viscosity of three emulsions was measured using a viscometer (Device Code: QC.L.05, Model: SVM3001) at the laboratory of the Ministry of Industry and Minerals, Industrial Research and Development Authority, Iraq. The measurements were carried out at a controlled temperature of 40°C. The viscosity reading was recorded after emulsions stabilization in order to evaluate the flow behavior of each formulation prior to spray drying.

2.5 | Microencapsulation Using Spray Drying Technology

Finally, emulsions were prepared and spray-dried using a spray dryer (ProCepT, Switzerland) at an inlet temperature of 180°C, an outlet temperature of 60°C–67°C, an air flow rate 40–42 m^3/min, a feed rate of 5.2 g/m^3, and a pump rate of 40%. The spray-dried emulsions were collected in tightly sealed amber glass bottles to protect it from light and oxidation (Kori et al. 2022) as shown Figures 1 and 2.

2.6 | Analysis of Microcapsule Properties

2.6.1 | Surface Morphology

A Quattro S-STEM/SEM scanning electron microscope was used to analyze powder models. The samples were coated with gold prior to analysis to obtain images with clear details of the particle surfaces.

2.6.2 | Size of Microcapsules

The particle size of the microcapsule was determined using a Master sizer 2000 (Malvern Instruments, UK), which measures particle size in both solid and liquid samples through laser light scattering, analyzing each powder sample three times to ensure accuracy.

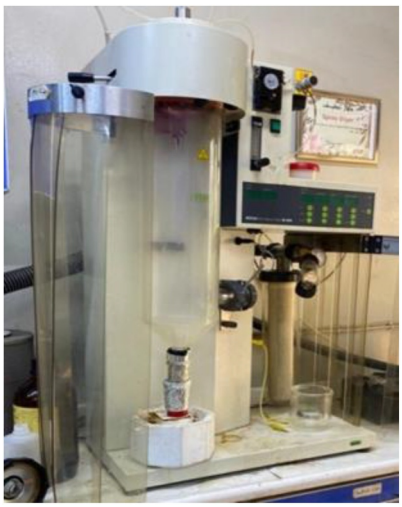

FIGURE 1 | The spray drying device used in the micro-encapsulation process.

TABLE 1 | Shows the ratio of wall materials to oil.

Lineage	Maltodextrin%	Concentrated milk protein%	Lecithin%	Water (mL)	PSO%
1:3 (A)	64.17%	7.50%	3.33%	350	25.00%
1:3.5 (B)	62.57%	8.38%	52.79%	325	26.26%
1.4 (C)	65.85%	7.32%	2.44%	300	24.39%

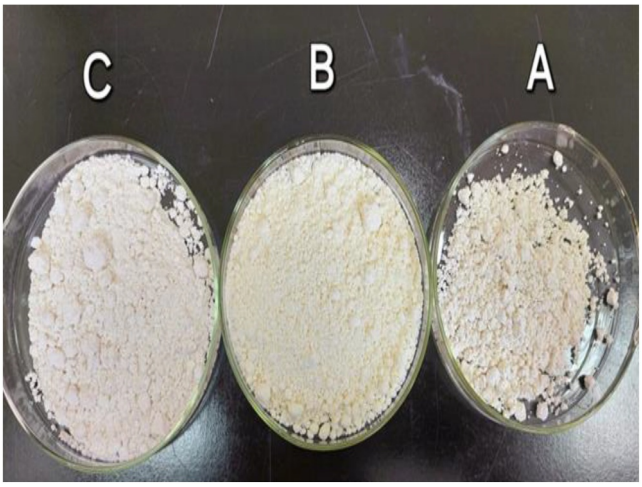

FIGURE 2 | Microcapsules of pomegranate seed oil produced by spray drying.

2.6.3 | Encapsulation Efficiency

Encapsulation efficiency is an important parameter that indicates effectiveness of the microencapsulation process in protecting the core material (in this study, pomegranate seed oil, PSO) within the wall matrix. It is defined as the proportion of the total oil that is successfully enclosed within the wall material relative to the total oil used in preparation. The oil that is not completely encapsulated remains on the surface of the microcapsules and is referred to as surface oil or free oil, which is more prone to oxidation during storage (Timilsena et al. 2020).

Mathematically, EE can be expressed as follows:

$$\mathrm{EE} = \frac{\text{Total oil} - \text{Surface oil}}{\text{Total oil}} \times 100$$

2.6.4 | Surface Oil Content

Surface oil content was determined using the method explained by Mohammed et al. (2021) with some modifications. 125 mL of 99% ethanol with 1.5 g of powder was stored in a tightly sealed glass bottle, and a vortex mixer was used to be shaken for 15 min at room temperature in the absence of light to obtain the free oil. The solvent mixture was filtered using No 4 filter paper, and the collected powder was then rinsed three times with 20 mL of ethanol. The filtrate, along with the extracted oil, was transferred to a rotary evaporator to evaporate the solvent; this was done at 30°C to avoid fat oxidation due to heating. The amount of surface oil was determined then calculated based on the extracted oil:

$$\text{Surface oil content} = \frac{W_1 - W_2}{W} \times 100$$

W_1 = Empty plate weight

W_2 = Weigh the dish with oil after drying

W = Sample weight (grams)

2.6.5 | Total Oil

PSO extraction from microcapsules was carried using an ultrasonic-assisted technique according to a modified method described by Gök et al. 2024. Five grams of encapsulated powder were placed in a beaker with 50 mL of 99% ethanol. The mixture was mixed using a magnetic stirrer for 15 min to ensure dispersion, then subjected to ultrasonic treatment in a water bath at 40°C for 10 min. Following sonication, the mixture was centrifuged at 3000–5000 rpm for 10 min to separate the extracted oil from the sediment, the solvent was then evaporated using a rotary evaporator at a low temperature to prevent oxidation of the oil.

2.6.6 | Bulk Density

The apparent density of the powder was measured by placing 2 g of powder inside a 10 mL graduated cylinder and subjecting the cylinder to repeated surface blows until a difference in volume was observed. The apparent mass was calculated as the mass ratio of the powder to the volume (Kori et al. 2022).

2.6.7 | Moisture Content

The moisture content of the micro-encapsulated powders was measured according to the method published by AOAC (2010). 3 g of powder were placed in an oven at 105° for a period of time until weight stability was achieved. The sample was then transferred to the dryer to cool. The weight of the sample was measured after the drying process. The experiment was repeated three times and the humidity was calculated according to the following equation:

$$\text{Moisture content (\%)} = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

W_1 = Empty plate weight

W_2 = Weight of the dish and sample before drying

W_3 = Weight of the dish and sample after drying

2.6.8 | Solubility

The solubility test was carried out according to Mohammed, Tan, et al. (2017). Except some modifications, 2 g of powder were placed in 100 mL of low temperature distilled water. The solution is mixed well for 10 min at 6000 rpm using a magnetic stirrer. After that, the solution is placed in a centrifuge at 3000 rpm for 5 min. 20 mL of the solution was transferred to a pre-weighed petri dish in a drying oven at 70°C until a stable weight separated the solids. The filtrate was then taken to a rotary evaporator to remove the solvent at a temperature of 40°C degrees. After obtaining the oil, it is kept in a tightly closed tube until tests are carried.

$$\text{Solubility} = \frac{\text{Weight gain}}{\text{Original weight}} \times 100$$

2.7 | Storage Stability

The storage stability of PSO microcapsules was studied at room temperature for periods of 0, 7, 14, 12, and 28 days. The effect of both temperature and exposure period on the peroxide number and antioxidant content of the coated powder was studied (Drinić et al. 2020).

2.7.1 | Storage Stability Analysis

2.7.1.1 | Extracting Oil From Microcapsules. The oil was extracted from the microcapsules using ultrasound using the same method used for total oil extraction when calculating encapsulation efficiency.

2.7.1.2 | Peroxide Value (PV). The peroxide value (PV) of the oil extracted from the microcapsules was evaluated based on Tavakoli et al. (2024) Using a modified method, the solvent mixture was prepared by mixing 25 mL of glacial acetic acid with 12.5 mL of chloroform in a 2:1 ratio. 12.5 mL of this mixture was then added to 2.5 mL L of oil sample, followed by 0.5 mL of saturated solution of potassium iodide (KI). The beaker was tightly closed and shaken for 2 min. After the reaction was complete, 15 mL of distilled water was added to wash away the free iodine adhering to the beaker walls. The liberated iodine was then titrated with 0.01 N sodium thiosulfate solution until the yellow color began to fade. At this point, a few drops of starch reagent were added, resulting in a blue color. The titration was continued until the blue color completely disappeared, indicating the end point. A similar titration procedure was performed without adding oil to determine the blank value. The volume of thiosulfate used for both the sample and blank was recorded, and the peroxide value was then calculated using the following equation:

The peroxide value (PV) = (mL of sodium thiosulfate consumed per sample-mL of sodium thiosulfate consumed for plank) × Standard × 1000/(Sample weight (grams)

2.7.1.3 | Antioxidant Activities by DPPH Methods. A free radical capture assay was used to evaluate the antioxidant capacity of the oil, according to the method described in Turki et al. (2024) with some modifications. A 0.004% solution of 1,1-diphenyl-2-picrylhydrazyl was prepared (using 4 mg of DPPH dissolved in 100 mL of 95% methanol). Three concentrations of sample and solvent (10, 15, and 20 μg) were prepared, to which 1 mL of solvent (ethanol or methanol) was added. Then, 0.5 mL of each concentration of the samples was taken, and 3 mL of DPPH solution was added to it. The samples were incubated for half an hour in the dark at room temperature. The absorbance was read at a wavelength of 517 nm against the absorbance of the control, which was prepared by taking 3 mL of DPPH solution and adding 0.5 mL of methanol solvent. The inhibitory effect was calculated according to the following equation:

free radical scavenging effect = (Control absorbance − Absorption of the mixture)/(Control absorbance) × 100

2.8 | Statistical Analysis

Data analysis was performed using SAS software, version 9.4., and one-way analysis of variance (ANOVA) was performed. Standard error (SE) was applied. The significance level ($p < 0.05$) was adopted to evaluate the differences.

3 | Results and Discussion

3.1 | Emulsion Characterization

3.1.1 | Emulsion Stability

The prepared emulsion must remain stable for a period after the drying process. In this study, three different emulsion ratios were prepared, each consisting of the packaging materials used to package PSO. The stability of the emulsions was evaluated 24 h after the homogenization process. The results indicated that all emulsions maintained their physical stability, with no layer separation, creaming, or oil leakage observed over the 24-h period. Our results were consistent with Comunian et al. (2020). Although different packaging materials were used, the emulsions showed good stability during the evaluation period. The selection of the type and proportions of packaging materials contributes mainly to maintaining the stability of emulsions (Wani et al. 2020).

3.1.2 | Viscosity

The results of the emulsion viscosity measurements showed a clear increase in viscosity with increasing encapsulating agent content, whereas the changes in oil content were slight. The viscosity values recorded were 3.81 cP for Emulsion A, 4.29 cP for Emulsion B, and 12.41 cP for Emulsion C, attributing to the increased amount of encapsulating agent. These results were consistent with those of many previous studies by Saxena et al. (2008). It has been reported that the concentration of coating materials, particularly proteins and polysaccharides, plays a major role in increasing the viscosity of emulsions due to the interpenetration of polymer molecules and water retention. These results differ from those of some studies, such as by Gharsallaoui et al. (2007), which have indicated that increasing the oil content directly leads to increased viscosity due to the increased size and cohesion of oil droplets within the emulsion. This difference is explained by the fact that the oil content in the B and A formulations is similar, whereas the coating material content is significantly higher in the C formulation, making the coating material concentration the dominant factor on viscosity. A study further supports this observation, showing that higher maltodextrin and protein contents increase the viscosity of emulsions despite stable or even low oil content Through our study, increasing the viscosity of the Emulsion C affected the spray drying process, as we noticed difficulty in the emulsion flowing through the drying tube, whereas in Emulsion A, the

emulsion flow was fast and would not affect the drying process, meaning that the ratio of A is considered the best in the drying process.

3.2 | Size of Microcapsules

The size of the microcapsules of the encapsulated oil should be less than 100 μm to ensure an acceptable taste of the food product, as the particle size is an important factor related to the quality and application of microcapsules (Kaushik et al. 2015). The particle size test of emulsions (A, B, and C) was performed using two methods: Surface Weighted Mean $D_{[3,2]}$ and Vol. Weighted Mean $D_{[4,3]}$ as shown in Table 2 Figure 2. Sample A showed the smallest average particle size of the two measurements, indicating good distribution of fine particles, which is desirable in food applications in terms of sensory acceptance and taste Sample B was close to Sample A, being within the acceptable range of 100 μm. Sample C recorded the highest average volume $D_{[4,3]}$ 94.60 μm, indicating large particles that negatively affect texture and taste. The results indicate that Sample A had the best particle size distribution due to the homogeneous distribution of the proportions of packaging materials to oil, as well as the appropriate homogenization time, which enhances the properties of the final product. Our results were comparable to those (Sahin-Nadeem and Afşin Özen 2014) of the post-encapsulation particle size (11.30–36.27 μm) using maltodextrin, modified starch, and whey protein as encapsulation materials (Comunian et al. 2020). The particle size ranged from (9.86–22.60 μm) when using whey protein, Gum Arabic, maltodextrin, and modified starch as wall materials, followed by the application of the spray drying process.

3.3 | Surface Morphology

A scanning electron microscope (SEM) study was conducted on the three emulsions, and the results revealed clear differences in the morphology of the microcapsules depending on the oil-to-wall material ratios as shown in Figure 3. For ratio A, the particles exhibited a regular spherical shape with a smooth surface, free of cracks or surface oil aggregates, reflecting high encapsulation efficiency. For ratio B, the results were similar to ratio A, with the capsules having a regular, spherical shape but containing a significant amount of surface oil. For ratio C, the particles were irregular, unevenly packed, and contained surface oil, reflecting a lower packing density. The results indicate that ratio A was the most efficient at capturing oil and thus achieving the highest packing efficiency. Our findings were consistent with those reported by Mangope et al. (2024) who found that the 1:3 coated particles exhibited smoother and more cohesive surfaces when both maltodextrin and Gum Arabic were used as the materials to form the capsule wall.

3.4 | Encapsulation Efficiency

Microencapsulation efficiency (MEE) is defined as the ability of the wall material to contain the matrix within a spherical structure, and the quality of microencapsulated oils depends largely on this efficiency of powders Mohammed et al. (2021) as shown in Table 3. The encapsulation efficiency ratio of the emulsion A is (91.60%), the surface oil ratio is (0.01%), and the total oil ratio is (0.11%). The high encapsulation efficiency ratio indicates a homogeneous distribution of the encapsulating materials. Also, the preparation conditions such as mixing speed and homogeneity were ideal, which led to the complete encapsulation of the oil and prevented the accumulation of oil on the surface. This result is considered ideal and effective in protecting the oil from oxidation and improving its stability. As for emulsion B, the encapsulation efficiency ratio was (69.96%), the surface oil ratio was (0.05%), and the total oil was (0.18%). The lower encapsulation efficiency compared to emulsion A is attributed to the higher surface oil content, which may be due to different wall material ratios or an increased oil content, which led to incomplete encapsulation and left some oil on the surface. In emulsion C, the encapsulation efficiency reached 55.56%, whereas the surface oil content was 0.02% and the total oil content was 0.04%. It was noted that the encapsulation efficiency decreased with the significant increase in the surface oil ratio compared to the total oil, indicating an inappropriate ratio of wall materials to the amount of oil. The results indicate that emulsion A has the best encapsulation efficiency compared to other ratios due to the homogeneous distribution of wall materials and ideal processing conditions. The use of a balanced three component coating mixture resulted in high coating efficiency and a reduction in surface oil indicating increased oxidative stability as observed in emulsion A. This is attributed to the synergistic interactions between maltodextrin, milk protein concentrate, and lecithin. Maltodextrin works by forming a hard coating around the oil droplets during drying, reducing the surface oil content, whereas milk protein concentrate reduces surface tension and stabilizes the emulsion by binding to the interface between the oil and water phases (Cevik et al. 2024). Lecithin, a phospholipid, enhances emulsion stability through its hydrophilic and hydrophobic properties (Mohammed et al. 2020). These complementary reactions result in a cohesive coating that reduces oil diffusion and protects the substrate from oxidation during spray drying.

Our results were consistent with those of Kaseke et al. (2025). The study demonstrated that using specific ratios of packaging

TABLE 2 | Particle size and viscosity of the three emulsions.

Emulsion	Surface weighted mean $D_{[3,2]}$	Vol. Weighted mean $D_{[4,3]}$	Viscosity (centipoise)
A	8.41 ± 0.07^{a} μm	11.66 ± 0.10^{a} μm	3.81 ± 0.07^{a}
B	15.54 ± 0.06^{a} μm	22.91 ± 0.05^{a} μm	4.29 ± 0.03^{a}
C	23.10 ± 0.06^{a} μm	94.60 ± 0.05^{a} μm	12.41 ± 0.04^{a}

Note: Data are expressed as means with standard deviation (±SD), whereas different lowercase letters indicate significant differences between values at a significance level ($p \leq 0.05$).

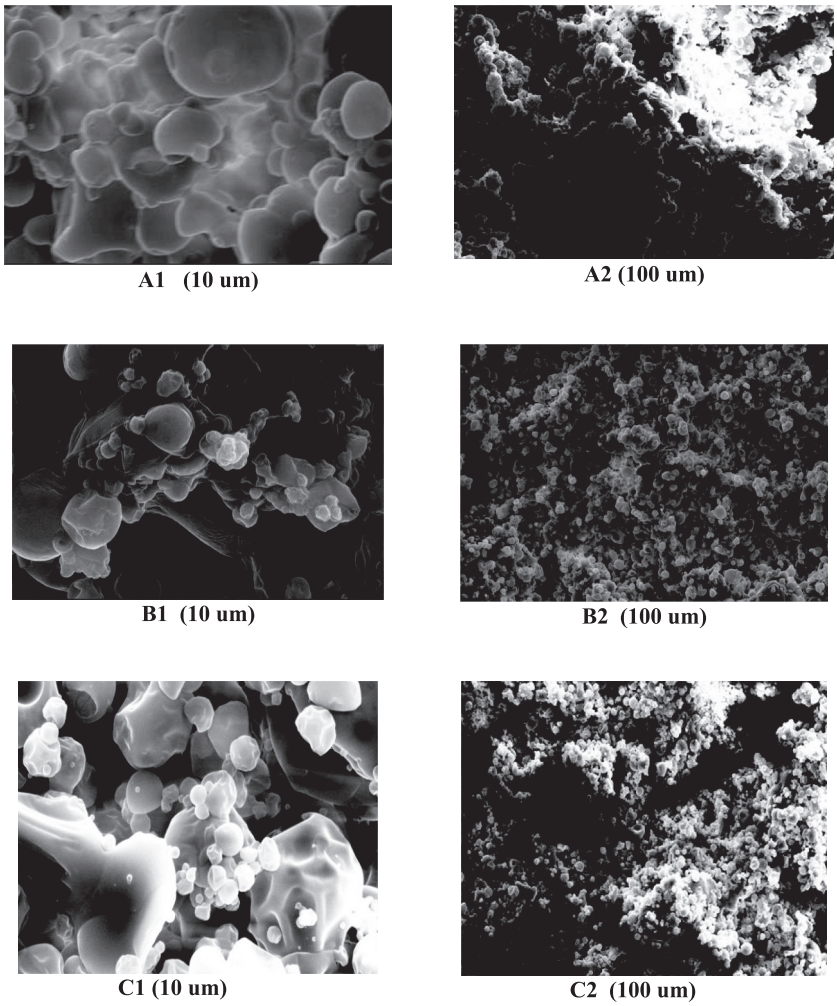

FIGURE 3 | The microstructure of emulsion A at 10 μm magnification (A1) and 100 μm magnification (A2), The microstructure of emulsion B at 10 μm magnification (B1) and 100 μm magnification (B2), The microstructure of emulsion C at 10 μm magnification (C1) and 100 μm magnification (C2).

TABLE 3 | Encapsulation efficiency, surface oil, solubility.

Emulsion	Encapsulation efficiency	Total oil	Surface oil
A	91.60 ± 1.18^{b}	0.11 ± 0.01^{a}	0.01 ± 0.00^{a}
B	69.96 ± 5.89^{b}	0.18 ± 0.01^{a}	0.05 ± 0.01^{a}
C	55.56 ± 9.62^{b}	0.04 ± 0.02^{a}	0.02 ± 0.01^{a}

Note: Data are expressed as means with standard deviation (±SD), whereas different lowercase letters indicate significant differences between values at a significance level ($p \leq 0.05$).

materials, such as maltodextrin and whey protein isolate, contributes to increasing packaging efficiency and reducing the amount of surface oil, which leads to enhanced stability of the packaged oil (Sahin-Nadeem and Afşin Özen 2014).

3.5 | Moisture Content

Moisture plays a critical role in evaluating quality and shelf life of the powder. Elevated moisture levels impair flow ability and promote stickiness of the particles as a result of crystallization and glass transition phenomena, causing them to clump and aggregate. This affects the properties of the microcapsules and makes them more susceptible to collapse and oxidation over time (Carneiro et al. 2013). The moisture content of the emulsion A is 2.33, which is a low percentage, indicating the efficiency of the drying process in removing water, which leads to reducing water activity and preventing the growth of microscopic organisms. This means the stability of the powder during storage. As for emulsion B, the moisture content was 2.96, which is close to the result of emulsion A, with a slight increase that may be due to a difference in the properties of the emulsion before drying. Emulsion C had a higher moisture content of 7.38 due to lower packaging efficiency or higher emulsion viscosity, which hinders water evaporation. High moisture content negatively affects the stability of the emulsion during storage,

leading to powder clumping and accelerating the oxidation and microbial decomposition process shown in the Table (4). These findings are consistent with those of (Sahin-Nadeem and Afşin Özen 2014) who recorded moisture content values of 2.3–3.5 g/100 g using a mixture of starch derivatives and whey protein isolate. Reducing the concentration of wall materials, increasing the oil percentage, and reducing the particle size leads to a decrease in the moisture level in the powder. Also, increasing the temperature of the air entering the spray drying device to 180 degrees Celsius leads to a decrease in moisture, meaning that the greater the temperature difference between the drying medium and the particles, the faster the heat is transferred to them (Goula and Adamopoulos 2012).

3.6 | Solubility

When studying properties of powder in aqueous medium, solubility is the most important and reliable parameter and is determined after performing the dissolution steps, which include immersion, wetting, and dispersion (Mohammed et al. 2021). The results showed that the solubility ratio in emulsion A reached 87.66, which is a high ratio indicating the ability to dissolve quickly in water and is suitable for food applications. As for emulsion B, the solubility ratio was 77.33, which is an average ratio. As for emulsion C, the solubility ratio was 78.33 as shown in Table 4. This indicates that the encapsulating materials are suitable for dissolving in water and that the distribution of the components is regular. Our results were consistent with those of Kori et al. (2022) which showed that the solubility percentage ranged from 89%–91% using whey protein alone or with maltodextrin as coating materials as explained by Comunian et al. (2020), The results showed similarity, with the solubility of the powder ranging from 88.16%–91.90%.

3.7 | Bulk Density

An important factor related to the packaging and marketing of powders is the bulk density used, as high density dry products can be stored in smaller packages compared to low density products (Quispe-Condori et al. 2011). The bulk density of the encapsulated PSO powder was 0.65 g/cm^3 in emulsion A, whereas in emulsion B it was 0.66 g/cm^3, and 0.68 g/cm^3 in emulsion C. The results were close. As shown in Table 4, our results were close to the study by Sahin-Nadeem and Afşin Özen (2014). It was shown that the apparent density was in the range of 0.262–0.522 g/cm^3 while using starch derivatives and whey protein as coating materials. A density of 0.22 g/cm^3 was obtained while mixing maltodextrin with whey protein at 125°C. At a higher temperature (150°C), the density increased to between 0.22 and 0.23 g/cm^3 (Kori et al. 2022). The significant increase in bulk density in our study can be attributed to the use of a mixture of encapsulating materials (maltodextrin, milk protein concentrate, and lecithin) in appropriate proportions along with optimal spray drying conditions, which resulted in more compact particles with high physical quality.

3.8 | Oxidative Stability

The most common methods for assessing oxidative stability are the peroxide value (PV) and free radical scavenging capacity (DPPH) and (TPC). PV reflects the concentration of primary peroxides produced by lipid oxidation, whereas DPPH indicates the antioxidant capacity responsible for preventing oxidative reactions. These two methods are commonly used to monitor and assess food quality (Mohammed, Meor Hussin, et al. 2017). Table 5 shows the evolution of PV and DPPH and total phenol content (TPC) in free oil and encapsulated oil every 7 days for 28 days at a temperature of 28°C. At the beginning of storage, the free oil showed PV 2.06 ± 0.01 (meq/kg), which was a slight difference from the encapsulated oil 2.00 ± 0.00 (meq/kg). The DPPH in the free oil was 91.75 ± 0.3% and in the encapsulated oil 91.51 ± 0.04%. The total phenolic content was 1.50 mg GAE/g before encapsulation and decreased slightly to 1.46 mg GAE/g after encapsulation, indicating a very slight decrease during the encapsulation process. We note an increase in the value of PV and DPPH after 28 days of storage in free oil and at a higher rate, as the PV ratio was 42.03 ± 0.02 (meq/kg), whereas in the encapsulated oil the ratio was 4.00 ± 0.05 (meq/kg) and DPPH 55.33 ± 0.40% for the free oil and 69.30 ± 0.09% for the encapsulated oil. The phenolic content in the free oil also decreased more rapidly, reaching 0.90 mg GAE/g, compared to the coated oil, which retained a higher percentage of 1.20 mg GAE/g, thus reinforcing the role of cell wall materials in improving oxidative stability. This indicates that the encapsulation process efficiently protects the oil from oxidation, compared to free oil. Our results were also similar to those of Yekdane and Goli (2019). The powder was stored at 25°C for 30 days, during which a peroxide test was conducted. The results showed the stability of the encapsulated oil. A study clarified (Kori et al. 2022). Capsules produced by spray drying technology using maltodextrin and whey protein demonstrated high resistance to oxidation, maintaining their oxidative stability at 60°C for 15 days, whereas the encapsulated oil showed lower oxidative activity compared to the free oil.

TABLE 4 | Moisture content, solubility, bulk density.

Emulsion	Moisture content (g/100 g)	Solubility%	Bulk density (g/cm^3)
A	2.33 ± 0.53^a	87.66 ± 0.57^a	0.65 ± 0.01^a
B	2.96 ± 0.97^a	77.33 ± 1.45^b	0.66 ± 0.00^a
C	7.38 ± 3.67^b	78.33 ± 1.20^b	0.68 ± 0.00^a

Note: Data are expressed as means with standard deviation (± SD), whereas different lowercase letters indicate significant differences between values at a significance level ($p \leq 0.05$).

TABLE 5 | DPPH and peroxide number (PV) results after oxidative stability of the coated powderv.

Days of treasury stability	(DPPH%) microencapsulation	(DPPH%) free oil	(PV meq/kg) microencapsulation	(PV meq/kg) free oil	(TPC mg GAE/g) microencapsulation	(TPC mg GAE/g) free oil
0	91.51 ± 0.04^{a}	91.75 ± 0.03^{a}	2.06 ± 0.00^{a}	2.10 ± 0.01^{a}	1.50 ± 0.02^{a}	1.58 ± 0.02^{a}
7	87.44 ± 0.03^{a}	83.57 ± 0.15^{a}	2.37 ± 0.15^{a}	12.05 ± 0.03^{a}	1.35 ± 0.01^{a}	1.28 ± 0.02^{a}
14	80.63 ± 0.06^{b}	76.53 ± 0.25^{b}	3.13 ± 0.15^{b}	22.05 ± 0.01^{a}	1.20 ± 0.01^{a}	0.95 ± 0.00^{a}
21	77.56 ± 0.08^{b}	67.53 ± 0.15^{a}	3.37 ± 0.15^{b}	32.06 ± 0.02^{b}	1.05 ± 0.03^{b}	0.78 ± 0.01^{b}
28	69.30 ± 0.09^{b}	55.33 ± 0.40^{b}	4.00 ± 0.05^{b}	42.03 ± 0.02^{b}	0.90 ± 0.00^{b}	0.57 ± 0.02^{a}

Note: Data are expressed as means with standard deviation (±SD), whereas different lowercase letters indicate significant differences between values at a significance level ($p \leq 0.05$).

4 | Conclusion

According to the results we obtained, microencapsulation was successful in encapsulating PSO from oxidation and improving its physical and chemical stability. Three emulsion ratios were tested and the ratio 1:3 showed the best properties regarding emulsion stability, ease of flow through the drying device, and particle size. The encapsulation efficiency was 91.60%, indicating high oil retention and a surface oil content of 0.01%, which reduces oxidation. The encapsulated powder showed a solubility of 87.66% and moisture content of 2.33 a bulk density of 0.65% g/mL, confirming its suitability for use in functional food for emulsions. Electron microscopy revealed that the capsules were spherical and free of cracks. Storage stability tests showed that the capsules retained 69.30% antioxidant activity and 0.90 mg GAE/g total phenol content (TPC) over 28 days and a peroxide number of 4.00 (meq/kg). This confirms the effectiveness of using maltodextrin, concentrated milk protein, and lecithin as wall materials in the spray drying encapsulation process. This study presents a promising approach for exploiting pomegranate seed by-products through green extraction and microencapsulation, which contributes to achieving sustainable development goals and expands the use of PSO in food and pharmaceutical applications.

Author Contributions

Hala Rabea Damen: conceptualization, investigation, formal analysis, writing – original draft, visualization, writing – review and editing. **Nameer Khairullah Mohammed:** supervision, methodology, project administration, writing – review and editing, funding acquisition. **Anis Shobirin Meor Hussin:** conceptualization, methodology, project administration, writing – original draft, investigation, writing – review and editing. **Belal J. Muhialdin:** formal analysis, writing – original draft, visualization. **Muammar Talib Hamad:** methodology, project administration, investigation, writing – review and editing. **Arpit Shrivastava:** methodology, project administration, writing – review and editing.

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The authors have nothing to report.

Consent

The authors have nothing to report.

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

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