***Original Research Article***

**Optimization of Spray Drying Parameters and Wall Material Composition for Stable Emulsion-Based Microencapsulation of *Hippophae rhamnoides* (Sea Buckthorn) Seed Oil**

**Abstract**

This study aimed to develop microencapsulated powders of *Hippophae rhamnoides* (sea buckthorn) seed oil with the help of maltodextrin and inulin as encapsulating wall materials via spray drying. The investigation stressed evaluating the impact of wall material composition and processing parameters on particle size, emulsion stability, encapsulation efficacy, moisture content, flowability, and structural characteristics of the microcapsules. Among various formulations, the optimal encapsulation conditions were identified at a maltodextrin-to-inulin ratio of 1:2, with a total wall concentration of 15%, a core-to-wall ratio of 1:3, and an inlet temperature of 160 °C. Given these optimized conditions, the formulation exhibited high encapsulation efficiency (94.94 ± 0.41%) and moderate process yield (56.91 ± 0.15%). The microcapsules showed a particle size distribution favorable for controlled release, an emulsion stability index of (92.43 ± 8.17%), and appropriate moisture content (3.89 ± 0.05%) and water activity (0.35 ± 0.003), suggesting good physical stability. Bulk density and tapped density were recorded as (0.31 ± 0.01 g/cm³) and (0.48 ± 0.01 g/cm³), respectively. However, flowability analysis revealed poor flow characteristics, with Carr’s index (34.74 ± 0.83) and Hausner ratio (1.52 ± 0.02), likely due to the high oil content (~36% w/w, dry basis). FTIR spectral analysis confirmed the successful encapsulation of seed oil through the identification of characteristic functional groups associated with both the core and wall materials. These findings highlight the critical role of wall material selection and process optimization in achieving high-quality microcapsules for potential application in functional food & nutraceutical formulations.

**Keywords:** Bulk density, Microencapsulation, Sea buckthorn seed oil,Encapsulation efficiency, Maltodextrin

**1-Introduction**

Herbal formulations have been utilized globally for centuries, serving both therapeutic and prophylactic purposes, as well as for promoting overall health. *Hippophae rhamnoides* L., also known as sea buckthorn. It belongs to the family Elaeagnaceae, and is a distinctive and highly valued plant that has garnered rising scientific interest in recent years because of its notable medicinal and nutritional attributes. This spiny, nitrogen-enriching deciduous shrub naturally grows in the cold, dry regions of Europe and Asia. Owing to its diverse bioactive properties, sea buckthorn has been cultivated in various regions worldwide for both nutritional and pharmaceutical applications (Ahani & Attaran, 2022). India possesses the most diverse germplasm resources for *Hippophae rhamnoides*, with the northern and southwestern regions exhibiting the greatest global concentration of this species.

The seeds of *Hippophae rhamnoides* fruit are non-edible and constitute approximately 23% of the total fruit weight (w/w), containing an oil content ranging from 10.0% to 16.3% (García, 2019). The oil extracted from *Hippophae rhamnoides* seeds is particularly rich in unsaturated fatty acids (UFAs), having approximately 71.2% to 76.0% of the total lipid content. The predominant UFAs include oleic acid (OA), linoleic acid (LA), and α-linolenic acid (ALA). Oleic acid, a monounsaturated fatty acid, has been associated with a reduced risk of cardiovascular diseases through its ability to lower serum triglyceride (TG) levels (Marchlewicz et al., 2024). Linoleic acid (LA) and α-linolenic acid (ALA) are polyunsaturated fatty acids classified as essential, as their synthesis is not possible endogenously and must be acquired through the diet. Linoleic acid has been shown to reduce serum cholesterol levels and inhibit the formation of arterial thrombi, contributing to the prevention of cardiovascular and cerebrovascular disorders (Wang et al., 2022). Alpha-linolenic acid (ALA) is crucial for the neurological and visual development of infants and children, and it also contributes to the regulation and maintenance of normal blood lipid profiles in adults (Syren et al., 2018; Van Der Wurff et al., 2020). The concentration and relative proportions of oleic acid (OA), linoleic acid (LA), and α-linolenic acid (ALA) serve as key parameters in assessing the nutritional quality of edible oils. In *Hippophae rhamnoides* seed oil, the typical ratio of OA to LA to ALA is approximately 1:4:2 (Solà Marsiñach & Cuenca, 2019).Besides unsaturated fatty acids, *Hippophae rhamnoides* seed oil is enriched with a variety of bioactive compounds, including carotenoids, vitamins, polyphenols, and flavonoids, which exhibit antioxidant, antibacterial, antiviral, and immunomodulatory activities (Bouras et al., 2017). These properties position *Hippophae rhamnoides* seed oil as a highly promising functional edible oil.

Due to its high unsaturated fatty acid content, *Hippophae rhamnoides* seed oil is prone to oxidative degradation and rancidity upon exposure to air, which results in a limited shelf life (Abd El-Aal et al., 2019). Oxidative processes negatively impact the organoleptic properties, diminish the bioactive efficacy, and lead to the formation of peroxides and free radicals, which have the potential to induce cellular damage and contribute to disease development (Huirong et al., 2021). Consequently, preventing the oxidation of *Hippophae rhamnoides* seed oil is critical to preserving its functional properties and ensuring its suitability for practical applications.

Microencapsulation technology effectively protects unsaturated fatty acids from oxidation by encasing oil particles within a polymeric shell. This protective barrier minimizes exposure to oxygen by reducing the interaction between the oil and air, thereby inhibiting oxidative degradation and prolonging oil stability (Singh et al., 2021).Spray drying is a commonly employed technique for microcapsule production due to its high throughput, effective encapsulation efficiency, uniform particle size distribution, and operational simplicity (Yang et al., 2024). The quality of microcapsules generated by spray drying is directly influenced by the conditions employed during both emulsion preparation and the spray drying process.

The primary objective of this research was to evaluate the effectiveness of microencapsulation in preserving the bioactivity of *Hippophae rhamnoides* seed oil. Additionally, the preparation parameters were optimized for the production of produce *H. rhamnoides* seed oil microcapsules using spray drying.

**2-Materials and Methods**

Sea buckthorn seed oil was acquired from Deve Herbs (Delhi, India).The material was subsequently encapsulated with the help of a core-to-wall material ratio of 2:1. All reagents and chemicals employed in the analysis were of analytical grade & procured from Hi Media Laboratories Pvt. Ltd., Mumbai, India.

**2.1-Manufacturing of microcapsules containing Sea Buckthorn seed oil**

Sea buckthorn seed oil microcapsules (SBSOM) were prepared with the help of a wall material composed of maltodextrin (MD) and inulin in a 1:2 ratio (Table 1). The microencapsulation was conducted via spray drying with the help of a mini spray dryer (JISL). The system was equipped with an atomizing nozzle featuring an internal diameter of 0.7 mm, and the feed flow rate was controlled by adjusting the pump rotation speed. The spray drying process was performed under the following conditions: inlet air temperature of 160–180 °C, outlet air temperature of 80–85 °C, a constant feed rate of 400 mL/h, air pressure maintained at 117.68 kPa, and vacuum pressure at 169.32 kPa. Before initiating the spray drying process, the equipment was operated for 30 minutes to ensure the attainment of steady-state conditions.

**2.2 Determination of encapsulation efficiency and yields**

Equal quantities of microcapsules were dispersed in hexane, with one sample subjected to shaking alone, while the other was both shaken and sonicated for one hour to acquire the respective supernatant solutions. The absorbance of each supernatant was calculated individually to determine the surface oil (SO) & total oil (TO) contents. Subsequently, encapsulation efficiency (EE) was calculated using the corresponding formula

EE=1-\*100

The microcapsules retained in the separator were collected, and the yield was determined using the following equation

Y=\*100

where M is the mass of the microcapsules and

m is the total mass of the seed oil and wall material

**2.2 Characterization of emulsion**

**2.2.1 Particle size and zeta potential**

The particle size and zeta potential of the emulsions were determined using a Zeta/Nanoparticle Analyzer (NanoPlus, Labindia Instruments Pvt. Ltd., Nano Bio Process Division, Thane, India) equipped with a dynamic light scattering system, as described by Chaudhary et al. (2020). For analysis, 1.5 mL of each emulsion sample was diluted in 5 mL of distilled water. Measurements of particle size and zeta potential were done separately and repeated until three consistent readings were recorded.

**2.2.2 Emulsion stability index (ESI)%**

To assess emulsion stability, 100 mL of each emulsion sample was transferred into a graduated cylinder and stored at 4 °C for 24 hours. Following the storage period, the volumes of both the separated and unseparated phases were recorded. Emulsion stability was then quantified as a stability index, expressed as a percentage

Emulsion stability index - \*100

Where,

H0 stands for the initial emulsion volume and

H1 stands for the unseparated phase volume of the emulsion (Binsi *et al.,* 2017).

**2.3 Bulk (ρB) and tapped (ρT) density**

To determine the bulk density (ρB), 2 g of each powder sample was transferred into a 25 mL graduated glass cylinder (2.5 cm diameter). The cylinder was gently tapped to dislodge any powder adhering to the walls, and the initial volume (Vo) was recorded. Bulk density was then calculated using given formula. For the measurement of tapped density (ρT), the same cylinder was tapped manually nearly 50 times on a solid marble surface from a height of 10 cm to achieve volume compaction. The final volume (Vn) was noted, and tapped density was calculated using given formula:

Bulk density=

Tapped density=

The flow characteristics of SBSOM were assessed using Carr’s Index (Compressibility Index, C) and the Hausner Ratio (HR), following the method described by (Turchiuli et al.2005). Carr’s Index measures the powder's compressibility and ability to flow freely, whereas the Hausner Ratio reflects the degree of cohesiveness among powder particles. Both indices were calculated based on the measured bulk and tapped densities using the given formulae, respectively

Carr index\*100

Hausner Ratio=

**2.4 Moisture Content and Water Activity (aw):**

The moisture content of sea buckthorn seed oil powder was analyzed using a halogen moisture analyzer (WENSAR, HMB 100, Bengaluru, India), operated under standardized conditions at 108 °C for 5 minutes. Each measurement was performed three times to guarantee precision and consistency of the results. The water activity (aw) of the microencapsulated functional oil powder (MSBSOP) was assessed at 35 °C using a water activity meter (AQUA Lab Pre, Decagon Devices, WA, USA). Prior to measurement, the instrument was adjusted using charcoal powder at the same temperature to ensure precise readings.

**2.5 Fourier transform infrared spectroscopy (FT-IR)**

Using the compression method, FT-IR samples were prepared. A measured amount of microcapsules was thoroughly mixed with 198.0 mg of KBr in a mortar, finely ground, and then compressed into transparent pellets. The spectra were noted with the help of a PerkinElmer Spectrum 3™ FT-IR spectrometer over the wavenumber range of 4000 to 600 cm⁻¹.

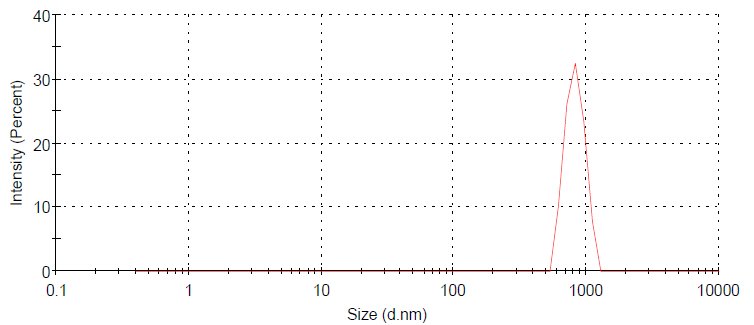
**2.6 Data analysis**

All data were collected from three independent replicates and are presented as mean ± standard deviation. Statistical analyses were performed using SPSS version 25.0 and Origin 9.0. Statistical significance between several groups was assessed using Duncan's multiple range test to determine meaningful differences.

**3 Results and Discussions**

**3.1 Effect of the preparation process on the particle size of microcapsules**

Particle size serves as a critical parameter for evaluating the quality of microcapsules. Smaller particle sizes facilitate more efficient release of the encapsulated core material (Nogueira et al., 2019). At lower concentrations of maltodextrin, insufficient emulsification and dispersion of *Hippophae rhamnoides* seed oil led to the formation of larger particles. Conversely, excessive maltodextrin levels increased the emulsion's viscosity, promoting particle agglomeration during the spray-drying process. Optimal particle size reduction was observed at a maltodextrin-to-inulin ratio of 1:2 (Figure 1).



**Figure 1- particle size distribution of microcapsules under optimal preparation process.**

**3.1.1Emulsion stability index**

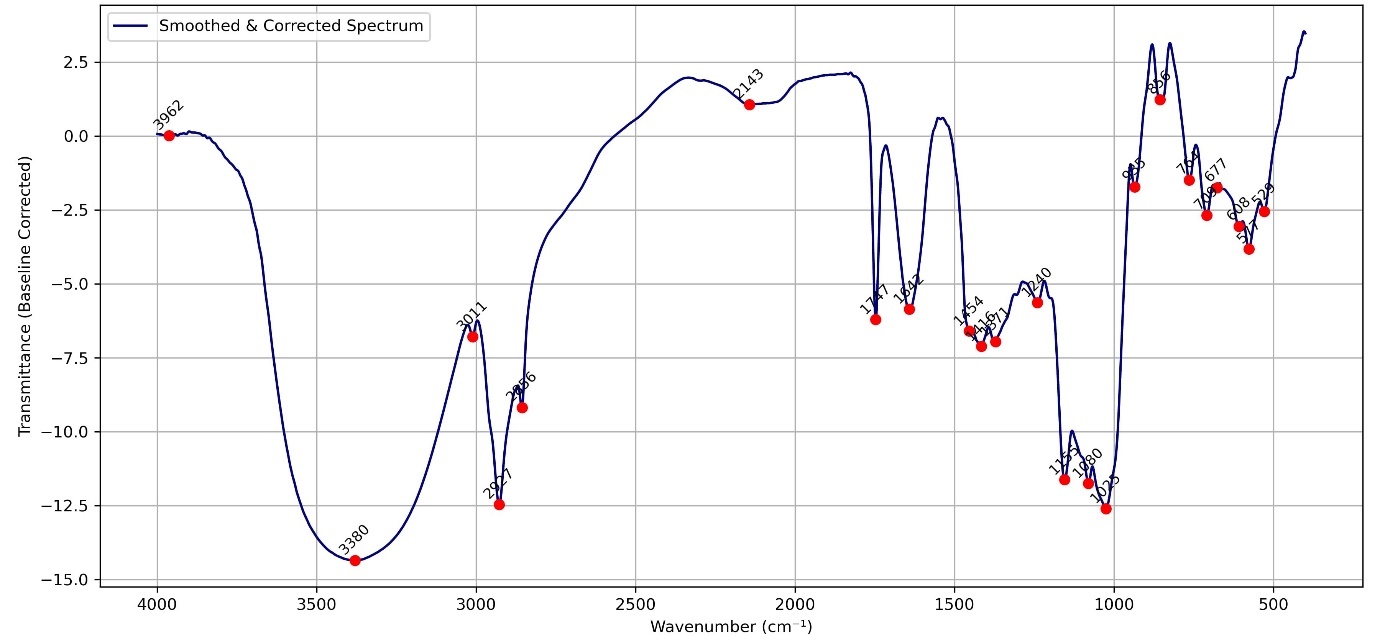
The emulsion stability index (%) was analyzed post-homogenization to find out the stability of the encapsulated *Hippophae rhamnoides* seed oil extract. Stability measurements were conducted at 24-hour intervals to monitor potential phase separation or degradation over time. (Cekić et al., 2023). The emulsion exhibited a stability index of (92.43 ± 8.17%), indicating a high degree of stability under the given conditions.

**3.2 Optimum Conditions of Microcapsules**

In accordance with encapsulation efficiency (EE) and yield results, the optimal formulation parameters were identified as a maltodextrin-to-inulin wall material ratio of 1:2, a wall concentration of 15.0%, a core-to-wall ratio of 1:2, and an inlet temperature of 160 °C. Under these conditions, the encapsulation efficiency of *Hippophae rhamnoides* seed oil reached (94.94±0.41) with a corresponding yield of (56.91±0.15).

**3.3 FT-IR analyses**

The FTIR spectrum (Figure 2) of the sea buckthorn seed oil microcapsules—processed with baseline correction and smoothing—reveals distinct transmittance bands corresponding to the characteristic functional groups present in both the core material (sea buckthorn seed oil) and the wall materials (maltodextrin and inulin). The spectral range analyzed spans from 4000- 400 cm⁻¹.A broad and intense absorption band centered at 3380 cm⁻¹ is attributed to the stretching vibrations of hydroxyl (–OH) groups, indicative of hydrogen bonding interactions within polysaccharide-based wall matrices such as inulin and maltodextrin. This also confirms the presence of moisture or polyol groups. The peaks observed at 2962 cm⁻¹, 2921 cm⁻¹, and 2856 cm⁻¹ correspond to the asymmetric & symmetric stretching vibrations of –CH₃ and –CH₂ functional groups, which features of long-chain aliphatic hydrocarbons present in fatty acid esters of the sea buckthorn oil. A moderate absorption at 2143 cm⁻¹ may be linked with the stretching of –C≡C– or –C≡N groups, which can be attributed to minor unsaturated or nitrile components within the oil composition. The sharp peaks at 1741 cm⁻¹ and 1701 cm⁻¹ are assigned to carbonyl (C=O) stretching vibrations. These are predominantly associated with ester groups found in triglycerides, a major component of the seed oil, and may also arise from residual free fatty acids or esters formed during spray drying. In the fingerprint region, a complex set of peaks is observed. Notably, 1544 cm⁻¹ and 1461 cm⁻¹ are indicative of C–H bending vibrations and possible amide II bands, potentially arising from protein residues or minor bioactive compounds present in the oil.The band at 1240 cm⁻¹ corresponds to C–O stretching vibrations, typical of ester and ether linkages, which may arise from both encapsulating agents and the oil itself. Multiple peaks between 1150 cm⁻¹ and 1050 cm⁻¹ further confirm the presence of glycosidic linkages (C–O–C and C–OH), representing the structural backbone of maltodextrin and inulin.The region between 900–500 cm⁻¹ shows out-of-plane C–H bending vibrations, which may be linked to substituted aromatic rings or skeletal vibrations from complex bioactive compounds, possibly flavonoids or phytosterols inherent to the seed oil.



**Figure 2-Fourier transform infrared spectroscopy (FT-IR) spectra of *H. rhamnoides* seed oil microcapsules**

**3.4-Moisture content and water activity (aw)**

Moisture content is a critical parameter in determining the shelf life of powdered products. Elevated moisture levels can promote fungal growth and induce caking, thereby negatively impacting the physical and chemical stability as well as the overall consumer acceptability of the product. Typically, a moisture content in the range of 3–4% is considered the minimum standard for most dried powders utilized in the food industry (Karaca et al., 2013).Moisture content of sea buckthorn seed oil powder (3.89±0.05).Generally, lipid oxidation in most dried food products is minimized at a water activity level ranging from (0.2 to 0.3). This oxidation reduction is primarily attributed to the diminished catalytic activity of transition metals, effective quenching of free radicals and singlet oxygen, and the inhibition of hydroperoxide decomposition under these conditions. In the current study (Table 2), the water activity (aw) of microencapsulated sea buckthorn seed oil powder (MSBSOP) was observed to be approximately (0.35±0.003). In general, moisture content and water activity (aw) are influenced by various factors, including the composition of core and wall materials, inlet and outlet temperatures, spray dryer flow rate, and the design of the drying equipment. In the present study, all processing parameters and ingredient quantities were maintained constant, except the type of wall material used. The findings of this study are consistent with the results reported by (Quispe-Condori et al., 2011) and (Aghbashlo et al., 2012), who observed moisture content values ranging from (3.88% to 5.06%) in flaxseed oil powder and from (1.41% to 4.36%)in fish oil powder, respectively.

**3.5 Bulk density (ρB) and Tapped density (ρT)**

Physical properties such as bulk density, tapped density, as well as compressibility significantly influence the flowability characteristics of powdered materials stability. In the present study, bulk density of MSBSOP formulations is (0.31±0.01g/cm3), which is the typical range of bulk density for microencapsulated powders (Onwulata & Holsinger, 1995 : Tuyen et al., 2014). These results indicate that the type of wall material used significantly influenced the bulk density of the resulting powders. Density is defined as mass divided by volume; thus, for a fixed mass, an increase in volume results in a decrease in density. Consequently, a comparable inverse relationship is expected between the bulk density of the powder and the particle diameter. The results obtained in this study are consistent with those reported by (Quispe-Condori et al., 2011) and (Tonon et al., 2012), who documented bulk densities for microencapsulated flaxseed oil powder ranging from (0.174 to 0.350 g/cm³) and (0.289 to 0.458 g/cm³), respectively.

The tapped density of a material affects critical aspects such as packaging efficiency, transportation, and the commercial handling of powders (Carr, 1965). It is evident from that the tapped density of MSBSOP formulation was found to be in the range of (0.48±0.01 g/cm3)(Table 2), The results indicated that the tapped density of the powders was significantly influenced by the type of wall material used. The results obtained align with those reported by (Finney et al., 2002), who recorded comparable tapped density values ranging from (0.48 to 0.65 g/mL) for microencapsulated orange essential oil powder (Finney et al., 2002).

**3.6 Flowing properties**

The flow characteristics of dry powders are commonly evaluated using Carr's index (percent compressibility) and the Hausner ratio. A compressibility value of approximately 20–25% typically distinguishes free-flowing (granular) powders from those that are non-free-flowing (Carr, 1965). In the present investigation (Table 2), it was discovered that the Carr's index for MSBSOP formulations ranged between (34.74±0.83), which corresponds to the reference values of 32–37 (Table 3), which revealed the very poor flowability of the developed MSBSOP formulations. It has been reported that the oil content within the powder adversely affects its bulk density, leading to a decrease in flowability (Sharma et al., 2012).In this study, all formulations of sea buckthorn seed oil powder exhibited a high oil content (~36% w/w, dry basis), which likely contributed to the reduced flowability of the prepared samples. Throughout this study, the sea buckthorn seed oil powder formulations consistently contained a high oil content (~36% w/w, dry basis), which is presumed to have negatively impacted the flowability of the resulting powders.(Quispe-Condori et al., 2011) documented poor flowability in flaxseed oil microcapsules, as evidenced by elevated Carr’s index values ranging from 33.72 to 48.65.Likewise, numerous studies have reported limited flowability in microencapsulated powders incorporating various types of oils (Turchiuli et al., 2005:Kagami et al., 2003).The Hausner ratio (HR) is a commonly used parameter to assess powder flowability. Elevated HR values signify increased powder cohesiveness and reduced flowability. Typically, a Hausner ratio exceeding 1.34 is indicative of poor flow characteristics in dry powders (Turchiuli ET AL., 2005). In the current study, HR for MSBSOP formulations was in the range of (1.52±0.02).

**4-Conclusion**

The present study successfully demonstrated the microencapsulation of *Hippophae rhamnoides* seed oil with the help of a spray drying technique with maltodextrin and inulin as wall materials. The optimized formulation, characterized by a 1:2 maltodextrin-to-inulin ratio, a wall concentration of 15%, and an inlet temp. of 160 °C, resulted in microcapsules with high encapsulation efficiency and satisfactory yield. The produced microcapsules maintained low moisture content as well as water activity levels, essential for extended shelf life and stability. Although the flow properties were suboptimal due to the high oil content, the physicochemical and structural analyses confirmed the successful entrapment of the oil within the matrix. FTIR spectroscopy validated the presence of functional groups representative of both wall and core materials, further supporting the encapsulation effectiveness. Overall, the findings underscore the importance of selecting appropriate wall material combinations and optimizing processing conditions to improve the quality, along with functionality of oil-based microcapsules. These microcapsules offer promising potential for use in functional food systems and nutraceutical applications, particularly where oil stability and controlled release are desired.

**Abbreviations**

MBSOP- Microencapsulated sea buckthorn seed oil powder

SBSOM-Sea buckthorn seed oil microcapsules

EE-Encapsulation efficiency

SO-Surface oil

TO-Total oil

CR-Carr’s Index

HR-Hausner Ratio

**COMPETING INTERESTS DISCLAIMER:**

Authors have declared that they have no known competing financial interests OR non-financial interests OR personal relationships that could have appeared to influence the work reported in this paper.

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| --- | --- | --- |
| **S.No** | **Wall material and proportion (A:B)** | **Ratio(A:B)** |
| 1 | Maltodextrin: inulin | 1:1 |
| 2 | Maltodextrin: inulin | 1:2 |
| 3 | Maltodextrin: inulin | 2:1 |
| 4 | Maltodextrin: gum Arabic | 1:1 |
| 5 | Maltodextrin: gum Arabic | 2:1 |
| 6 | Maltodextrin: gum Arabic | 3:1 |
| 7 | Maltodextrin: sodium caseinate | 1:1 |
| 8 | Maltodextrin: sodium caseinate | 2:1 |
| 9 | Maltodextrin: sodium caseinate | 3:1 |

**Table 1- Selection of appropriate combinations and ratios of wall materials**

|  |  |
| --- | --- |
| **Parameters** | **Values** |
| Moisture content (%) | 3.89±0.05b |
| Water activity (aw) | 0.35±0.003b |
| Bulk density (ρB) (g/cm3) | 0.31±0.01b |
| Tapped density (ρT) (g/cm³) | 0.48±0.01b |
| Carr's index(CI) | 34.74±0.83b |
| Hausner ratio (HR) | 1.52±0.02b |
| Encapsulation efficiency (EE%) | 94.94±0.41b |
| Yield % | 56.91±0.15a |
| Emulsion stability index (ESI) % | 92.43±8.17b |

**Table 2-Physicochemical Characterization and Encapsulation Efficiency of Microencapsulated Sea Buckthorn Seed Oil Powder Using Optimized Wall Material Combinations. Values are presented as mean ± standard deviation (n = 3). Different superscript letters (a, b) within the same column that shows significant differences (p < 0.05)**

|  |  |  |
| --- | --- | --- |
| **Carr's index** | **Flowability** | **Hausner ratio** |
| Less than or equals to 10 | Excellent | 1.00–1.10 |
| 11.00 to 15.00 | Good | 1.12–1.19 |
| 16 to 20 | Fair | 1.19–1.26 |
| 21 to 25 | Passable | 1.26–1.35 |
| 26 to 31 | Poor | 1.35–1.45 |
| 32 to 37 | Very poor | 1.45–1.59 |
| Less than or equals to 38 | Awful | ≥1.60 |

**Table 3- Powder flowability from the Carr's index and Hausner ratio (Turchiuli et al., 2005).**