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Microencapsulation of Saffron Petal Extract Using Sodium Caseinate: Physicochemical Properties

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Abstract

Saffron, as one of Iran's strategic products, has stigmas and petals rich in phenolic compounds and anthocyanins, which can serve as a natural source of color and antioxidants. However, anthocyanins are unstable compounds that degrade rapidly under environmental factors such as light, heat, and oxygen. This study aimed to improve the stability and efficacy of bioactive compounds in saffron petals by microencapsulating them with sodium caseinate, enabling their application in dairy products. The saffron petal extract was prepared and, after concentration, mixed with the wall material, sodium caseinate, at different core-to-wall ratios (1:3, 1:5, and 1:7), and microencapsulated using the freeze-drying method. The control sample included the unencapsulated extract. Then, the physicochemical properties of the powders, including anthocyanin content, moisture, water activity, solubility, density, and flowability indices, were evaluated. The results indicated that the 1:7 ratio exhibited the best characteristics, including greater anthocyanin stability, reduced moisture and water activity, improved flowability, and increased solubility.

Keywords: Saffron petals, Anthocyanin, Microencapsulation, Sodium caseinate, flowability

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Introduction

Saffron petals are a by-product of saffron, with annual production exceeding 10,000 tons (Kafi & Showket, 2006). Compared to saffron stigmas, saffron petals are inexpensive and are commercially neglected. Saffron petals are a rich source of bioactive compounds, containing various flavonoid compounds (kaempferol, rutin, quercetin, luteolin, and hesperidin), tannins, anthocyanins, and their glycosides (Rahaiee et al., 2015). They contain 10.2% protein, 3.5% fat, 7% ash, 8.8% fiber, and essential minerals (calcium, potassium, and phosphorus) (Bakhshi et al., 2022), and possess numerous therapeutic properties, including sedative effects (Hosseinzadeh & Ghaenati, 2006), blood pressure–lowering effects, antidepressant activity (Akhondzadeh Basti et al., 2007; Moshiri et al., 2006; Hadizadeh et al., 2003), reduction of liver toxicity (Iranshahi et al., 2011; Omidi et al., 2014), weight reduction (Mohaqiq et al., 2020), and alleviation of premenstrual symptoms in women (Agha-Hosseini et al., 2008). Consequently, they reduce the risk of cardiovascular diseases, cancer, and stroke. Researchers have attributed the therapeutic properties of saffron petals to their flavonoid compounds, which are abundant and possess antioxidant properties.

Microencapsulation is a technique for packaging sensitive components and compounds within a coating or wall material to protect them from environmental physicochemical factors, oxidation, and evaporation. Using this method, in addition to preventing the loss of volatile and environmentally sensitive compounds, they can be released under controlled conditions. Therefore, active, sensitive, or volatile compounds such as vitamins, plant extracts, flavor- and aroma-generating compounds, essential oils, and others can be converted into stable forms using this technique (Korhonen, 2002). In this process, the coated materials are referred to as the core, active material, or internal phase, while the coating materials are known as wall materials, carriers, external phase, shell, or encapsulating agents (Zuidam & Nedovic, 2009). In the food industry, microencapsulation is used for various reasons, including reducing the reactivity of core materials with environmental conditions (such as light, heat, oxygen, and moisture), preventing their degradation, enabling gradual release over time or at a specific site, and masking undesirable odors or flavors of the core materials (Feng & Bhandari, 2010).

Freeze-drying is a process used to dry materials and essential oils that are sensitive to heat. Although time-consuming for microencapsulation, freeze-drying is a suitable and simple method for microencapsulating various water-soluble essential oils and natural aromas, including pharmaceuticals (Feng & Bhandari, 2010).

Sodium caseinate contains both hydrophobic and hydrophilic regions. This amphiphilic structure underlies its ability to emulsify, foam, and form films (Huppertz, Fox, & Kelly, 2018). Due to its emulsifying properties, film-forming ability, good solubility, and stability, sodium caseinate is one of the most suitable wall materials for microencapsulation in the food industry, especially for oily or environmentally sensitive compounds (Goyal & Sharma, 2011).

Feizy & Reyhani (2016) determined the phytosterols and fatty acids of saffron petals using gas chromatography. Dabbagh Moghaddam et al. (2018) produced a fermented probiotic saffron beverage using saffron petals and compared its physicochemical, antioxidant, rheological, and sensory properties with those of a simple non-fermented saffron beverage. Ahmad et al. (2018) microencapsulated saffron petal anthocyanins using spray drying with beta-glucan and beta-cyclodextrin as wall materials. Ahmadian et al. (2019) investigated the effects of spray-drying and freeze-drying methods and different wall structures (maltodextrin and pectin) on the physicochemical properties, morphology, particle size, stability, and release of phenolic content from saffron petal extract microcapsules in a simulated gastrointestinal system. Ghanbari et al. (2019) examined the characteristics of volatile organic compounds in saffron stigmas and petals from different corms grown under different dietary regimes and in different origins. Hashemi & Jafarpoor (2020) produced biodegradable edible films using konjac glucomannan and saffron petal extract and evaluated their effects on the quality and shelf life of fresh-cut cucumbers. Alizadeh-Sani et al. (2020) synthesized a novel halochromic film by entrapping saffron petal extract in biopolymer composite films made of chitosan nanofibers and methylcellulose using a casting method, and measured their optical, mechanical, barrier, thermal, spectroscopic, release, ammonia sensitivity, antioxidant, and antimicrobial properties. Pasban Noghabi & Saberian (2023), in a study entitled "Thermal stabilization of saffron petal anthocyanin extract using microencapsulation and its application in a food model," investigated strategies to enhance the stability of anthocyanins extracted from saffron petals. Sahel et al. (2024) investigated the anthocyanin content of saffron petals. Kermani et al. (2024) conducted a study entitled "Microencapsulation of saffron extract in liposomal nanocarriers and evaluation of their physicochemical properties." Gull et al. (2024) conducted a study to evaluate the stability and release of anthocyanin extract from saffron petal waste. Alipour et al. (2025) microencapsulated bioactive compounds of saffron extract within wall coatings of starch extracted from saffron corms at different levels using spray drying. Zarei et al. (2025) evaluated the effect of saffron extract microencapsulation on the quality and shelf life of Taftoon bread.

Numerous studies have been conducted on the characteristics of bioactive compounds in saffron petals, as well as their extraction and microencapsulation methods.

The innovative aspects of the study include:

Valorization of saffron petals, a low-cost and abundant by-product rich in anthocyanins and phenolic compounds.

Optimization of sodium caseinate ratios to enhance anthocyanin retention, solubility, flowability, and moisture stability.

Demonstrating that sodium caseinate can form effective protective matrices for highly unstable anthocyanins extracted from saffron petals.

Providing physicochemical data that support the potential use of the resulting powder as a natural colorant and functional ingredient in food systems.

The aim of this study is the optimal utilization of saffron by-products and the microencapsulation of saffron petal extract as a novel approach to innovation in producing a high-value-added saffron-based product, using sodium caseinate as an accessible and low-cost material, to increase anthocyanin stability against environmental conditions, and ultimately using it as a coloring and medicinal agent in food products.

Materials and Methods

Materials

Saffron flowers were harvested in November 2024, from selected farms in Torbat-e Heydarieh County, Razavi Khorasan Province, Iran. After harvesting, different parts of the flowers were separated, and the petals were dried at the Arnika processing plant (Torbat-e Heydarieh, Iran).

Sodium caseinate (Sigma–Aldrich, CAS No. 90005-46-3) was used as the wall material for the microencapsulation of saffron petal extract. All chemicals and solvents used in this study were of analytical grade.

Preparation of Saffron Petal Powder

The dried saffron petals were ground using a laboratory grinder and passed through a 16-mesh sieve. The resulting powder was placed in dark, airtight containers and stored at 4 °C until further analysis.

Preparation of Saffron Petal Extract

Ten grams of saffron petals were accurately weighed (±0.01 g) and transferred into a dark glass bottle. Then, 150 mL of 50% (v/v) ethanol, previously adjusted to pH 3 using 1.5 N hydrochloric acid, was added. The mixture was stirred continuously for 24 h in a dark environment at ambient temperature (25 °C).

After extraction, the mixture was filtered under vacuum using Whatman No. 1 filter paper. The residue was re-extracted with an additional 150 mL of the same solvent. The pH of the combined extracts was adjusted to 3 using 0.1 N hydrochloric acid to minimize thermal degradation.

The solvent was removed using a rotary evaporator at 40 °C for 60 min to concentrate the extract (Jafari et al., 2016). The concentrated extract was subsequently freeze-dried at −86 °C and 5 mmHg for 72 h using a freeze dryer (Operan, Korea).

Microencapsulation of Saffron Petal Extract

Microencapsulation was carried out using sodium caseinate as the wall material and saffron petal extract as the core material. Wall-to-core ratios of 3:1, 5:1, and 7:1 were prepared. The mixtures were stirred on a magnetic stirrer for 20 min to ensure homogeneity. The pH of the resulting emulsions was adjusted to 2 using 1.5 N hydrochloric acid to enhance anthocyanin stability.

Drying of Microencapsulated Samples

The prepared microencapsulated samples were freeze-dried at −86 °C under 5 mmHg for 72 h. The obtained solid material was immediately ground using a porcelain mortar, sieved through a 40-mesh sieve, and stored in airtight dark glass containers at −18 °C until analysis.

Preparation of the control sample

The concentrated saffron petal extract without any wall material was used as the control sample. The extract was freeze-dried under the same conditions as the microencapsulated samples (−86 °C, 5 mmHg, 72 h). The resulting porous solid was ground, sieved through a 40-mesh sieve, and stored in dark glass containers at −18 °C until further analyses.

Materials and Methods

Determination of Total Anthocyanin Content

Total anthocyanin content was determined using the pH-differential method described by Lee et al. (2008). Results were expressed as milligrams of cyanidin-3-glucoside equivalents per gram of dry powder.

Moisture Content

Moisture content was measured using a vacuum oven (Memmert, model 40050-IP-20, Germany) at 70 °C and 200 mbar for 24 h. After drying, samples were cooled in a desiccator containing silica gel at room temperature (Fang & Bhandari, 2011). Moisture content was calculated gravimetrically.

Water Activity

Water activity (aw) of the microencapsulated powders was measured immediately after production using a water activity meter (Lab Master aw, Mova Sina, Switzerland) at 25 ± 0.1 °C.

Bulk and Tapped Density

Bulk density was determined by gently filling 10 ± 0.001 g of powder into a 10 mL graduated cylinder and recording the occupied volume. Tapped density was measured after tapping the cylinder 20 times on a laboratory bench. Both densities were expressed as g/cm³ (Sharifi et al., 2015).

Flowability and Cohesiveness

Powder flowability was evaluated using the Carr Index (CI), calculated according to Equation (1) (Jinapong et al., 2008):

$$\text{CI}\ (\%) = (\text{Tapped density} - \text{Bulk density}) / \text{Tapped density} \times 100 \quad (1)$$

Powder cohesiveness was assessed using the Hausner ratio (HR), calculated using Equation (2):

$$\text{HR} = \text{Tapped density} / \text{Bulk density} \quad (2)$$

Statistical Analysis

All experiments were conducted in triplicate using a completely randomized design. Data were analysed by one-way analysis of variance (ANOVA) using SPSS software (version 20). Mean comparisons were performed using Duncan's multiple range test at a significance level of 5% ($p < 0.05$). Graphs were prepared using Microsoft Excel (version 2013).

Results and Discussion

Anthocyanin Content of the Powder after Production

Anthocyanins are inherently unstable compounds and are highly susceptible to degradation by light, heat, and oxygen. Therefore, microencapsulation within protective wall materials is crucial for preserving these pigments.

The effect of different levels of sodium caseinate (wall material) on the anthocyanin content of microencapsulated saffron petal powder is shown in Table 1. As observed, increasing the core-to-wall ratio significantly increased the anthocyanin content ($p < 0.05$).

Table 1-Effect of different core-to-wall ratios on anthocyanin content of microencapsulated saffron petal extract (mg.100 Ml^{-1})

Core-to-wall ratios	Anthocyanin content (mg.100 Ml^{-1})
0	1569.69۴ ± 23^{A}
3.1	946.836 ± 48^{C}
5.1	1446.122 ± 96^{B}
7.1	1553.596 ± 34^{A}

Different letters indicate significant differences at 95% confidence level. Values are mean ± SD (n=3).

The anthocyanin content of saffron petals was measured using the pH-differential method and was found to be 1569.6 mg cyanidin-3-glycoside per 100 mL of extract. This value is higher than that reported for other natural sources such as cranberry juice, red wine, and natural colorants (Jafari et al., 2016). Jafari et al. (2016) and Alizadeh Sani et al. (2020) reported anthocyanin contents of 1.71 and 1.994 mg/mL for saffron petals, respectively. These differences may be attributed to genetic variations. Given the high anthocyanin content of saffron petals compared to other sources, they represent a highly suitable raw material for anthocyanin extraction and industrial applications.

The significant differences in anthocyanin content between microencapsulated samples at core-to-wall ratios of 1:5 and 1:7 and the control sample indicate the effectiveness of microencapsulation in stabilizing pigments under the tested storage conditions. This confirms the protective role of the wall material against thermal degradation of anthocyanins. Since oxygen presence intensifies other degradation factors, and the combined effect of heat and oxygen is among the most destructive factors for anthocyanins (Jackman et al., 1987), the physical barrier formed by wall materials acts as a protective shield against moisture and oxygen.

Increasing the sodium caseinate concentration can result in thicker, more resistant walls. Due to its proteinaceous nature, sodium caseinate can form weak interactions with anthocyanins, reduce wall permeability, and consequently prevent anthocyanin leakage. Therefore, at optimal concentrations, sodium caseinate enhances microencapsulation efficiency and improves anthocyanin stability during storage and processing. However, excessive sodium caseinate may increase solution viscosity and reduce effective anthocyanin encapsulation by interfering with capsule formation or incomplete dispersion.

Moisture Content

In microencapsulated powders, an optimal moisture level (neither too high nor too low) is desired depending on the intended application. Excessive moisture may lead to instability, stickiness, or degradation, whereas overly low moisture may cause brittleness or premature release of active compounds. Therefore, the optimal moisture content depends on the active compound, the wall material, and the final application.

The effect of sodium caseinate as a wall material on moisture content depends on its physicochemical properties, the wall-to-core ratio, the microencapsulation technique, and the processing conditions. Statistical results (Table 2) showed that increasing the wall material ratio significantly decreased moisture content ($p < 0.05$).

Table 2- Effect of different core-to-wall ratios on moisture content (%) of microencapsulated saffron petal extract

Core-to-wall ratios	Moisture content (%)
0	7.16 ± 0.04^a
3.1	5.30 ± 0.01^b
5.1	4.66 ± 0.28^b
7.1	3.76 ± 0.08^c

Different letters indicate significant differences at 95% confidence level. Values are mean ± SD (n=3).

Higher sodium caseinate levels led to reduced final moisture content due to increased wall thickness and reduced moisture exchange with the environment. Sodium caseinate generally results in lower residual moisture compared to wall materials such as maltodextrin, owing to its protein structure and lower hygroscopicity. Additionally, sodium caseinate forms uniform, resistant films that limit water vapor penetration and enhance thermal stability during freeze-drying.

Interactions between sodium caseinate and the phenolic compounds in saffron petal extract resulted in stable matrix structures and protein–polyphenol complexes, reducing water vapor permeability and improving drying efficiency, thereby significantly lowering the final moisture content. Compared with polysaccharide wall materials such as maltodextrin and gum Arabic, sodium caseinate typically produces powders with lower moisture content (Farcas et al., 2018).

Water Activity

Statistical analysis of the effect of different wall material ratios (sodium caseinate) on the water activity (aw) of microencapsulated saffron petal powder is presented in Table 3. As the wall material ratio increased, aw significantly decreased ($p < 0.05$). As shown in the table, increasing the sodium caseinate concentration reduced water activity; however, this effect depends on processing conditions and the overall composition of the microencapsulation system. Water activity is influenced not only by total moisture content but also by the distribution of moisture (free and bound water) and the drying method employed. Sodium caseinate is a water-soluble protein containing polar functional groups such as $-COOH$ and $-NH_2$, which are capable of forming hydrogen bonds with water molecules. Through these interactions, free water is converted to bound water, reducing water activity. Moreover, increasing the concentration of the wall material (e.g., sodium caseinate) raises the total solid content of the system, thereby diluting the water relative to the formulation and leading to a decrease in aw, even when the absolute moisture content does not change substantially. Protein-based wall materials, such as sodium caseinate, can also reduce the equilibrium relative humidity, which is directly related to water activity. The results indicated that sodium caseinate, particularly at optimal concentrations, significantly reduced the water activity of microencapsulated saffron petal powder by forming a uniform matrix and hydrogen bonding with water molecules. This reduction plays a critical role in improving powder stability and shelf life. Although sodium caseinate is hydrophilic and capable of retaining water, excessive concentrations can increase aw; however, at lower concentrations or when combined with wall materials such as maltodextrin or gum Arabic, it can effectively reduce water activity. Saffron petals are rich in phenolic compounds, flavonoids, and anthocyanins. These compounds can interact with active groups (e.g., amino groups) in the sodium caseinate structure via non-covalent or hydrogen-bonding interactions. Such interactions result in a more stable wall structure, reduced water accessibility to the core material, and, consequently, lower water activity. This phenomenon, known as protein–polyphenol interaction, has been widely reported in the literature for its effectiveness in reducing water activity (Fang & Bhandari, 2020). When proteins such as sodium caseinate are combined with saffron petal extract, particularly extracts with high antioxidant capacity and reactivity, the resulting structure tends to retain a greater proportion of moisture as bound water (Rohn et al., 2002). Furthermore, the formation of a uniform and impermeable coating through interactions between caseinate and saffron bioactive compounds enhances the dispersion of the core material within the wall solution and promotes the formation of particles with more homogeneous coatings. This limits moisture transfer from the environment into the core and reduces aw. Previous studies have shown that polysaccharide-based wall materials generally reduce water activity more directly by forming drier, less permeable, and less hydrophilic structures. Comparative evaluations of various wall materials have demonstrated that polysaccharides, such as maltodextrin and gum Arabic, effectively reduce the water activity of microencapsulated powders (Fazeli et al., 2012; Jafari et al., 2008).

Table 3- Effect of different core-to-wall ratios on water activity (aw) of microencapsulated saffron petal extract

Core-to-wall ratios	Aw
0	0.205 ± 0.005^{a}
3.1	0.166 ± 0.007^{b}
5.1	0.156 ± 0.006^{bc}
7.1	0.133 ± 0.008^{c}

Different letters indicate significant differences at 95% confidence level. Values are mean ± SD (n=3).

Bulk Density and Tapped Density

In evaluating the physical properties of microencapsulated powders, bulk density and tapped density are two important, complementary parameters. Bulk density is the ratio of powder mass to its volume in a free-flowing, uncompacted state, whereas tapped density is measured after controlled tapping to reduce volume and increase particle packing. These two parameters differ considerably in both measurement and application, with tapped density generally being higher than bulk density. In microencapsulated powders, understanding and controlling these parameters can improve coating quality and process performance (Mohammad et al., 2015).

Tapped density is recognized as a key indicator in assessing the physical properties of microencapsulated powders. This parameter reflects the compressibility of particles under tapping or vibration and directly affects the final volume and compactness of the product. Tapped density can enhance powder flowability and reduce blockage problems in industrial processes, particularly in dosing and packaging operations (Mohammad et al., 2015). Moreover, tapped density is closely related to the Hausner ratio and can provide insight into particle aggregation and coating uniformity. Consequently, controlling and optimizing tapped density is crucial for improving the quality and performance of microencapsulated powders.

The Carr index and Hausner ratio are among the most important indices used to evaluate powder flowability and cohesiveness. These indices are also widely applied to investigate the physical behavior of microencapsulated powders after coating.

The Carr index is calculated from the difference between bulk and tapped density and is generally indicative of the energy required for particle size reduction and powder rearrangement. In microencapsulated powders, this parameter is particularly important because energy consumption influences coating distribution and adhesion on particle surfaces. A lower Carr index indicates easier grinding and mixing, leading to more uniform coatings, whereas a higher Carr index indicates greater cohesiveness and poorer flowability. If microencapsulation causes particles to adhere to one another or form agglomerates, the Carr index increases. Conversely, coatings that smooth the particle surface or reduce internal friction can decrease this index (Bond, 1961).

The Hausner ratio is calculated as the ratio of tapped density to bulk density and is an indicator of powder flowability. Higher Hausner ratios indicate greater interparticle adhesion and poorer flow behavior. In microencapsulated powders, a lower Hausner ratio corresponds to improved flowability and reduced problems such as clogging or particle agglomeration. This parameter is particularly useful in quality control and the design of packaging systems. If coating increases surface roughness or stickiness, the Hausner ratio will increase, whereas appropriate coatings, such as dry coatings with silica or magnesium stearate, can reduce it (Hausner, 1967).

Both the Carr index and Hausner ratio are rapid and effective tools for the preliminary assessment of powder flow quality and particle structure after microencapsulation. Evaluating these indices alongside analyses such as particle size distribution and moisture content provides a comprehensive understanding of the behavior of microencapsulated powders.

In powder microencapsulation processes, controlling the physical and mechanical properties of particles is essential to achieve uniform and efficient coatings. The Carr index is a key parameter whose optimization can enhance coating uniformity and reduce energy consumption during processing (Bond, 1961). Conversely, the Hausner ratio serves as an important measure of powder flowability. Lower values indicate improved flow characteristics and reduced issues such as particle aggregation and blockage in industrial processes (Hausner, 1967). Therefore, optimizing both parameters is critical for improving the quality of the final product and the overall efficiency of the process.

In microencapsulated powders prepared with various wall materials, such as gum Arabic, alginate, maltodextrin, and starch, the Carr index and Hausner ratio are key parameters for evaluating the physical performance of the powders.

Statistical results regarding the effect of different wall material ratios (sodium caseinate) on the Carr index and Hausner ratio, along with bulk and tapped density values of microencapsulated saffron petal powder, are presented in Table 4.

Bulk density showed a statistically significant difference only at the wall ratio of 1:5 compared with the other samples ($p < 0.05$). Tapped density values differed significantly among the tested samples ($p < 0.05$).

The flowability results based on the Carr index indicated that samples with wall-to-core ratios of 1:3 and 1:7 exhibited excellent flowability (Carr index < 15). No statistically significant difference was observed between these two ratios ($p > 0.05$). In contrast, the control sample and the 1:5 wall-to-core ratio sample showed higher Carr index values, indicating good flowability (15 < Carr index < 20). These samples differed significantly from the 1:3 and 1:7 samples ($p < 0.05$).

Table 4- Effect of different core-to-wall ratios on bulk density, tapped density, Carr index, and Hausner ratio of microencapsulated saffron petal extract

Core-to-wall ratios	Bulk density (g.cm^{-3})	Tapped density(g.cm^{-3})	Carr index (%)	Hausner ratio
0	0.677 ± 0.04^{a}	0.801 ± 0.03^{a}	15.401 ± 7.67^{a}	1.186 ± 0.11^{a}
3.1	0.699 ± 0.03^{a}	0.780 ± 0.02^{a}	10.347 ± 2.34^{b}	1.115 ± 0.03^{a}
5.1	0.582 ± 0.07^{a}	0.717 ± 0.02^{b}	18.636 ± 12.21^{a}	1.243 ± 0.19^{a}
7.1	0.689 ± 0.02^{a}	0.798 ± 0.02^{a}	13.636 ± 0.00^{b}	1.157 ± 0.00^{a}

Different letters indicate significant differences at 95% confidence level. Values are mean ± SD (n=3).

The cohesiveness index calculated from the Hausner ratio revealed a statistically significant difference between the control, 1:3, and 1:7 samples compared with the 1:5 sample ($p < 0.05$), while no significant differences were observed among the control, 1:3, and 1:7 samples themselves ($p > 0.05$). The aforementioned samples (control, 1:3, and 1:7) exhibited Hausner ratios below 1.20, indicating low cohesiveness, whereas the 1:5 sample showed a Hausner ratio above 1.20, corresponding to moderate cohesiveness (1.20–1.40).

The use of sodium caseinate as a wall material for microencapsulation of saffron petal extract resulted in improved Carr index and Hausner ratio values, reflecting favorable flow characteristics of the final powder. This improvement can be attributed to several factors. Sodium caseinate is a surface-active protein that forms a uniform and stable coating around extract particles, improving particle shape and surface smoothness while preventing adhesion and agglomeration. As the wall coating becomes more homogeneous, interparticle friction and adhesion are reduced, leading to improved flowability and directly lowering the Carr index and improving the Hausner ratio.

Due to its emulsifying properties and strong film-forming ability, sodium caseinate promotes the formation of near-spherical particles. Spherical particles exhibit minimal resistance to flow and readily slide over one another. In addition, sodium caseinate may contribute to a narrower particle size distribution, enhancing particle packing and reducing interparticle voids, which positively influences the Hausner ratio. As a wall material, sodium caseinate demonstrates good thermal resistance and structural stability during freeze-drying, preventing particle deformation or degradation and thereby improving particle structural quality (Ardestani et al., 2024).

Therefore, the use of sodium caseinate as a wall material in microencapsulation results in powders composed of spherical particles with smooth surfaces, higher bulk density, and improved flowability, ultimately leading to reduced Carr index and Hausner ratio values (Rajabi et al., 2015).

Flowability is a critically important property of microencapsulated powders in manufacturing processes, particularly in the pharmaceutical, food, and advanced materials industries (Owasit et al., 2025). Flowability refers to the ability of a powder to move and flow freely under the influence of gravity or vibration. In microencapsulated powders, this property is strongly affected by coating structure, particle size, surface adhesion, and moisture content (Hare et al., 2024).

Uniform powder flow is essential in tablet compression and capsule-filling equipment. Any blockage or flow fluctuation can result in tablets with non-uniform weight or composition. Powders with poor flowability tend to bridge or clog in hoppers and transfer channels, leading to process interruptions or reduced productivity. Inadequate flowability can also lead to an uneven distribution of the active ingredient, compromising the safety and efficacy of the final product. Consequently, good flowability is considered a key indicator of coating uniformity and overall powder quality from a quality control perspective (Tharanon et al., 2024).

Microencapsulation may reduce particle surface roughness, thereby decreasing interparticle friction and improving flowability. However, if the coating material is sticky (e.g., hydrophilic polymers), particles may adhere to one another, reducing flowability. Properly and uniformly coated particles exhibit superior flow behavior, whereas rough or agglomerated coatings can promote particle clustering and blockage. Some wall materials are hygroscopic and may induce particle agglomeration, thereby impairing flowability. In addition, microencapsulation can alter electrostatic charges on powder surfaces, thereby further enhancing interparticle adhesion and reducing flowability. Electrostatic effects may also influence powder behavior.

Cohesiveness is another key property of microencapsulated powders that influences their rheological behavior and, in turn, flowability, processability, and final product quality. Cohesiveness refers to the internal adhesive forces between powder particles that cause them to stick together or form agglomerates. In microencapsulated powders, cohesiveness is affected by surface coating properties, particle size, moisture content, and electrostatic charge.

Polymeric or adhesive coatings such as HPMC, PVP, and ethyl cellulose can increase surface stickiness and, consequently, powder cohesiveness. In contrast, some coatings may act as surface lubricants, reducing cohesiveness. Moisture absorption by the coating can promote particle adhesion and agglomerate formation. Finer particles, owing to their higher specific surface area, generally exhibit greater cohesiveness, whereas larger or more uniform particles tend to show lower cohesiveness. During the coating process, friction may generate electrostatic charges that cause particles to attract each other. Furthermore, if the coating is non-uniform or if certain areas remain uncoated, some particles may become excessively adhesive.

Powders with high cohesiveness typically exhibit poor flowability and tend to clog in hoppers or filling equipment. Particle adhesion leads to the formation of heterogeneous clusters within the final formulation, which may result in inaccurate dosing or non-uniform distribution of the active ingredient. Excessive cohesiveness can also disrupt uniform compaction, producing tablets with variable hardness or porosity. Highly cohesive powders are prone to caking during storage, thereby limiting their ease of reuse.

Microencapsulated powders prepared with gum Arabic generally exhibit Carr index values of 14-20 $kWh.t^{-1}$ and Hausner ratio values of 1.18-1.28, indicating moderate grinding energy requirements and acceptable flowability (Patel et al., 2016). Powders coated with alginate show higher Carr index values (approximately 18–25 $kWh.t^{-1}$) and Hausner ratio values between 1.25 and 1.38, attributable to the complex molecular structure and higher adhesiveness of this material (Gómez-Mascaraque et al., 2017). Maltodextrin, as a coating material, typically yields lower Carr index values (12–18) and Hausner ratio values (1.15–1.25), reflecting better flowability and lower grinding energy requirements (Sánchez et al., 2018). Starch-coated powders generally exhibit Carr index values of 15–22 and Hausner ratios of 1.20–1.30, indicating a favorable balance between flowability and compressibility (Martínez et al., 2019).

These differences in powder physical properties arise from variations in the structural and chemical characteristics of the coating materials, which ultimately influence the quality and performance of microencapsulated powders.

Solubility of Microencapsulated Powder

In microencapsulated powders, changes in solubility depend on the product's intended application. In general, when the goal is to enhance bioavailability or accelerate drug absorption, increased solubility is desirable. In such cases, microencapsulation protects the active compound (e.g., a drug) while enhancing solubility by selecting appropriate wall materials, such as water-soluble coatings or surfactants (Goyal et al., 2015). Conversely, when the objective is controlled release or protection of the active compound against environmental conditions (e.g., during gastrointestinal transit or storage), reduced or regulated solubility is preferred. In these cases, wall materials are selected to dissolve under specific conditions, such as at a particular pH or after a defined time interval, enabling gradual release of the active compound (Agyare et al., 2022). Therefore, increased solubility is advantageous for improving absorption, palatability, rapid dissolution, and bioavailability, whereas reduced solubility or slow release is beneficial for protection, targeted delivery, and reduction of side effects. Statistical analysis of the effect of different wall material ratios (sodium caseinate) on the solubility of microencapsulated saffron petal extract powder is presented in Table 5.

Table 5- Effect of different core-to-wall ratios on solubility of microencapsulated saffron petal extract

Core-to-wall ratios	Solubility (%)
0	21.95 ± 0.07^a
3.1	21.45 ± 0.35^a
5.1	22.7 ± 0.57^a
7.1	22.95 ± 1.13^a

Different letters indicate significant differences at 95% confidence level. Values are mean ± SD (n=3).

Increasing the wall-to-core ratio significantly increased solubility ($p < 0.05$). As shown in the table, no statistically significant difference was observed between the control sample and the core-to-wall ratio of 1:3. Similarly, the solubility values of the 1:5 and 1:7 core-to-wall ratios did not differ significantly at the 5% level ($p > 0.05$). Although these two ratios (1:5 and 1:7) did not show statistically significant differences from each other, they exhibited higher solubility values compared with the control and 1:3 samples. The results demonstrated that sodium caseinate, when used as a wall material in the microencapsulation process, significantly enhanced the water solubility of the core material. Due to its amphiphilic nature (both hydrophilic and lipophilic), sodium caseinate acts as an effective emulsifier, is highly soluble in water, and readily forms stable solutions. It can therefore maintain hydrophobic compounds (such as oils or poorly soluble bioactive compounds) in stable suspension or emulsion within aqueous systems (Lu et al., 2022). Consequently, the improved solubility of the microencapsulated saffron petal extract powder facilitates better dispersion of the active compounds in water.

Conclusion

Saffron petals are a major by-product of saffron processing and represent a rich source of bioactive and antioxidant compounds. This study aimed to microencapsulate saffron petal extract using sodium caseinate as a wall material. The use of sodium caseinate during the microencapsulation process significantly improved the physical and functional properties of the final powder. By forming a uniform, stable coating around the particles, sodium caseinate enhanced powder flowability by reducing both the Carr index and the Hausner ratio, indicating decreased internal friction and improved powder handling characteristics. In addition, sodium caseinate limited moisture exchange and promoted the formation of a denser structure, thereby reducing moisture content and water activity and enhancing the microbial stability of the powder. Furthermore, the application of this wall material improved anthocyanin retention during drying and storage. Owing to the favorable capsule structure, the solubility of the powder was also markedly improved. Considering the obtained values for moisture content, water activity, anthocyanin retention, powder solubility, as well as the calculated Carr index and Hausner ratio for the control sample and different core-to-wall ratios, and taking into account that the final microencapsulated powder should exhibit good flowability and low cohesiveness to ensure proper dispersion in the final product, along with high anthocyanin content, low moisture and water activity, and adequate solubility, the core-to-wall ratio of 1:7 was selected as the optimal formulation.

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