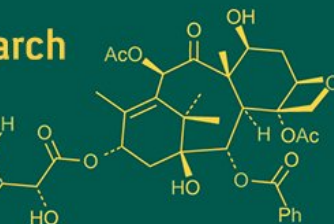
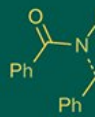


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Physico-Chemical and structural characterisation of foam-mat dried jamun (*Syzygium cumini* L.) Powder

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Abstract

Jamun (*Syzygium cumini* L.) is an anthocyanin-rich but highly perishable fruit and foam-mat drying offers a gentle, low-cost route to convert its pulp into a stable powder. This study presents a physico-chemical and structural characterisation of foam-mat dried jamun powder prepared with soya protein isolate (SPI) as the foaming agent and carboxymethyl cellulose (CMC) as the foam stabiliser, comparing a high-SPI formulation (5% SPI, 2% CMC) with a low-SPI formulation (1% SPI, 2% CMC). Particle size analysis (dynamic light scattering/zeta sizer), zeta potential measurement, scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), Fourier-transform infrared (FTIR) spectroscopy, and Raman spectroscopy were used to probe particle architecture, colloidal stability, surface morphology, elemental composition and molecular-level interactions. The high-SPI powder formed smaller, more uniform particles (Z-average $\approx$ 624 nm) with a compact, angular SEM morphology and fewer, sharper FTIR and Raman bands, indicating a well-cross-linked, stable protein-polysaccharide matrix. The low-SPI powder produced larger, more heterogeneous particles (Z-average $\approx$ 1071 nm) with a porous, open microstructure but a markedly higher zeta potential magnitude, reflecting greater electrostatic (colloidal) stability. EDS confirmed both powders were dominated by carbon and oxygen, consistent with their organic, fruit-derived composition and freedom from contamination. Together, these results show that SPI concentration governs the particle-scale architecture, surface charge and molecular organisation of foam-mat dried jamun powder, information that is directly relevant to its stability and functional performance as a food ingredient.

Keywords: Jamun; foam-mat drying; particle size; zeta potential; SEM-EDS; FTIR; Raman spectroscopy

Introduction

Jamun (*Syzygium cumini* L.) is a subtropical fruit that comes under myrtaceae family originated from the Indian subcontinent, but its high moisture content and thin skin make it extremely perishable seasonal fruit, resulting in a very short postharvest life and significant losses if not processed promptly. Hence processing and preservation are essential to ensure the availability of products throughout the year.^[10] Converting jamun pulp into a shelf-stable powder is a practical way to preserve its bioactive value and broaden its use as a functional ingredient in beverages, bakery products, dairy analogues and confectionery. Foam-mat drying is a process of drying a semisolid or liquid foods by incorporating with the foaming agent and foaming stabilizers in order to produce a stable foam, which is further air-dried at a temperature ranging from 50-80 °C.^[6] The foam-dried product will be further ground to produce a powdered product. It is the simplest form of drying when compared to other drying methods such as freeze drying and, spray drying as it is less expensive, less complicated and also less time consuming.^[5] This technique produces a final product that is porous, of superior quality and capable of regaining its original characteristics. Foam-mat drying is highly efficient, cost-effective and particularly suitable for heat-sensitive food materials because of its rapid drying process, minimal thermal degradation.^[7]

Dynamic light scattering (particle size and zeta potential), scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS), Fourier-transform infrared (FTIR) spectroscopy and Raman spectroscopy together provide a multi-scale picture of this architecture: particle size and zeta potential describe the dispersed-particle behaviour and

surface charge; SEM-EDS reveals surface morphology and elemental composition [12] and FTIR/Raman spectroscopy probe the functional groups and molecular interactions (particularly hydrogen bonding and protein-polysaccharide cross-linking) that underlie the physical structure.

The objective of this paper is to present a physico-chemical and structural characterisation of foam-mat dried jamun powder using zeta sizer-based particle size and zeta potential analysis, SEM-EDS, FTIR and Raman spectroscopy and to relate the structural differences observed to the underlying soya protein isolate concentration used during foam-mat drying.

2. Materials and Methods

2.1 Raw material and foam-mat drying

Fully matured, ripe jamun fruits were washed, deseeded and

pulped, and the pulp was filtered through muslin cloth to obtain clear juice. Soya protein isolate as a foaming agent and carboxymethyl cellulose as a foam stabiliser were incorporated into the juice and the mixture was whipped for a fixed duration of 5 minutes to generate a stable foam. The foam was spread as a thin layer (7-8 mm) onto trays and dried in a hot-air tray dehydrator at 60 °C for 5 hours, after which the dried foam sheet was scraped off and milled into a fine powder.

For detailed physico-chemical and structural characterisation, two contrasting treatments from Table 1. were selected to isolate the effect of the foaming agent while the stabiliser level was held constant: a high-SPI formulation (5% soya protein isolate, 2% carboxymethyl cellulose; T1) and a low-SPI formulation (1% soya protein isolate, 2% carboxymethyl cellulose; T5).

Table 1: Treatment runs of foam-mat drying of Jamun powder

Treatment runs	Soya protein isolate (%)	Carboxy methyl cellulose (%)
T1	5	2
T2	5	1
T3	3	3
T4	1	3
T5	1	2
T6	1	1
T7	3	2
T8	5	3
T9	3	1
T10	3	2

2.2 Particle size and zeta potential

Particle size was determined using a Zetasizer (Nano ZS, Model 383, Malvern, UK) based on dynamic light scattering (DLS). Powder samples were dispersed in 5 mL ethanol and ultrasonicated at 50 °C for 15 minutes to ensure uniform dispersion. The suspension was transferred to a cuvette (filled to three-fourths capacity) and analysed under standard instrument settings, with the average particle size (nm) obtained from the intensity-based size distribution [9]. Zeta potential was determined from particle mobility measured at multiple applied voltages, with the average value recorded from the corresponding intensity distribution [13].

2.3 Scanning electron microscopy-energy dispersive x-ray spectroscopy (sem-eds)

Surface morphology of the T1 (5% SPI, 2% CMC) and T5 (1% SPI, 2% CMC) foam-mat dried jamun powder samples was examined using a scanning electron microscope (Carl Zeiss Microscopy, EVO 10, Oberkochen, Germany). Samples were mounted on aluminium stubs (~1 cm dia.) cleaned with polyvinyl alcohol and compressed air to remove surface oils and dirt, and fixed using double-coated conductive carbon tape. The mounted samples were sputter-coated with gold-palladium for 90 s (Quorum Technologies, Q150R S, United Kingdom) to render them conductive, then imaged at an accelerating voltage of 10 kV with a working distance of 10-15 mm, adjusted according to sample size [5]. Images were captured at multiple locations on each sample to obtain a representative view of surface morphology and samples were compared and analyzed its elemental composition in the EDS.

2.4 FTIR spectroscopy

Functional group analysis of both samples was carried out using an FTIR spectrophotometer (IR Tracer 100 AH, Shimadzu, Japan) operated with LabSolutions software (Version 100). Spectra were acquired in percent transmittance mode over a scan range of 7800-350 cm⁻¹, with 45 scans per sample at 4 cm⁻¹ resolution, using a mid-IR beam splitter and a DLATGS detector for both sample and background scans. Approximately 0.5 g of jamun powder (sieved through 420 µm) was placed on the Specac Quest Diamond ATR attachment, and the top screw head was tightened to ensure uniform sample spread across the ATR compartment before scanning. Functional groups were identified by matching the resulting spectra against the LabSolutions-IR library using a peak-to-peak search, with the working wavenumber range (400-4000 cm⁻¹) selected following the procedure of [1].

2.5 UV-Visible spectroscopy

UV-Visible spectroscopy measures the extinction (absorption plus scattering) of light through a sample, a property sensitive to nanoparticle size, shape, concentration, agglomeration state and surface refractive index, making it a useful tool for characterising nanomaterials. Jamun powder was characterised using a UV-Visible spectrophotometer (PerkinElmer, Lambda 35, Germany). Aqueous sample solutions were filled to three-fourths of a quartz cuvette (1 cm path length), with ethanol as the reference. Absorbance was recorded over 350-410 nm using a double-beam spectrophotometer, and λ_{max} was determined from the resulting spectrum [2].

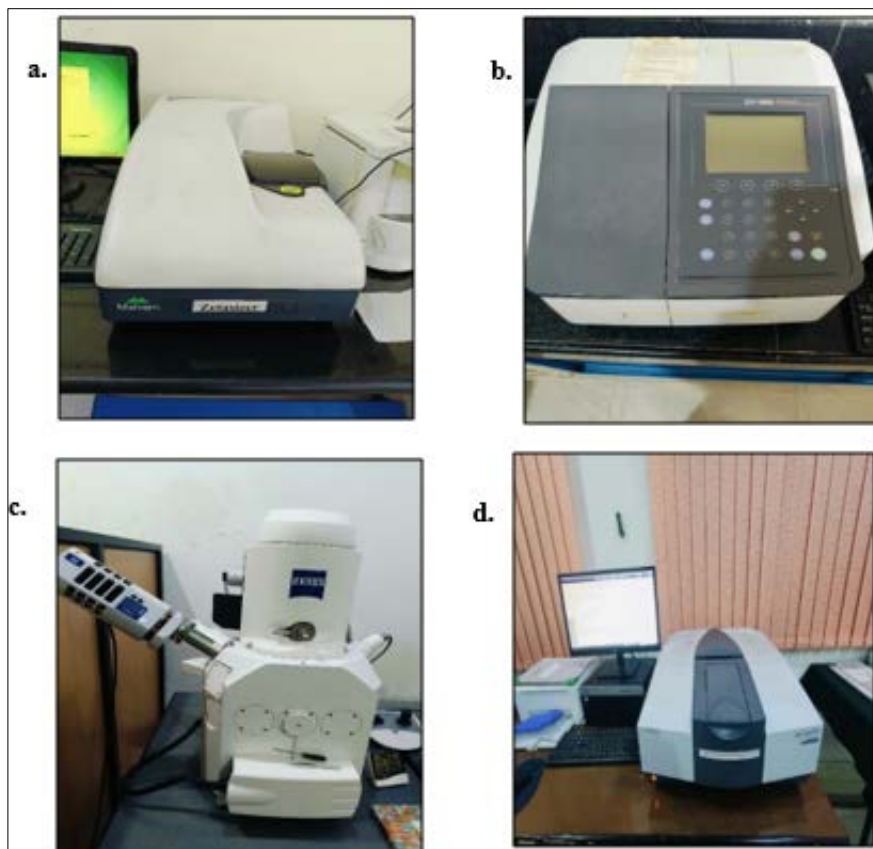


Plate 1: a) Zeta sizer b) UV-VIS spectrometer and c) Scanned electron microscopy (SEM) d) Fourier Infrared spectrum

3. Results and Discussion

3.1 Particle size distribution

The particle size distribution analysis of foam-mat dried jamun fruit powder revealed differences between treatments (Fig.1). Treatment T₁ with 5% soya protein isolate + 2% CMC showed a smaller Z-average particle size of 624.3 nm, with a bimodal distribution featuring peaks at 458.1 nm (76.2% intensity) and 98.47 nm (23.8% intensity) as observed in Fig 1a. In comparison, treatment T₅ with 1% soya protein isolate + 2% CMC had a larger Z-average of 1071 nm, with a trimodal distribution displaying peaks at 1666 nm (34.0% intensity), 316.1 nm (27.9% intensity), and 5172 nm (18.1% intensity). The polydispersity index was 1.000 for both treatments. T₁ with 5% SPI demonstrated better light scattering properties, with an intercept value of 0.925, compared to 0.665 for T₅. The smaller, more uniform particle population obtained at the higher SPI level is consistent with the strong emulsifying and film-forming action of soya protein isolate, which lowers interfacial tension during foam formation and limits particle agglomeration during drying and subsequent milling. Similar findings were reported in bottle gourd powder [11]

3.2 Zeta potential

Zeta potential, an index of the electrostatic stability of a particle dispersion, showed an inverse relationship with SPI concentration. The fig.2, represents zeta potential analysis of foam-mat dried jamun fruit powder which showed differences between the two treatment formulations. The low-SPI formulation (T₅) displayed a substantially higher zeta potential magnitude (≈ -28.1 mV, with markedly lower dispersion conductivity), whereas the high-SPI formulation (T₁) showed a much lower magnitude (≈ -4.95 mV) with higher conductivity. A larger zeta potential magnitude generally denotes stronger electrostatic repulsion between particles and therefore greater colloidal stability.

This can be attributed to lower SPI concentration in T₅, which reduces protein surface coverage and allows more negative charges from CMC and native fruit acids to dominate, whereas higher SPI in T₁ may lead to charge shielding and reduced surface potential. The lower conductivity in T₅ (0.729 mS/cm) versus T₁ (3.84 mS/cm) further supports reduced ion mobility and enhanced dispersion stability [3]

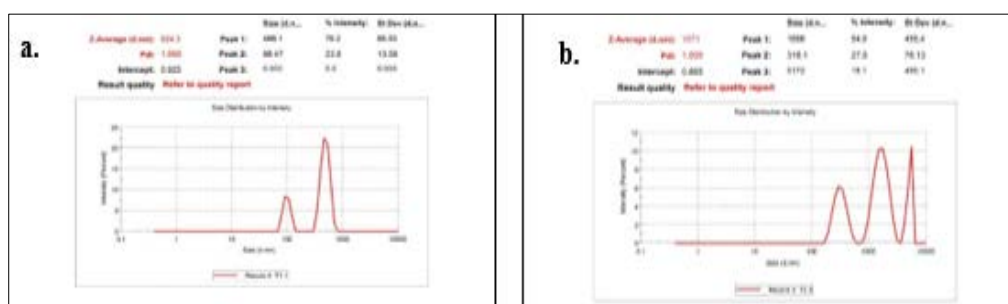


Fig 1: Particle size distribution of foam-mat dried jamun powder a) T1 (5%SPI, 2%CMC) b) T5 (1% SPI, 2%CMC)

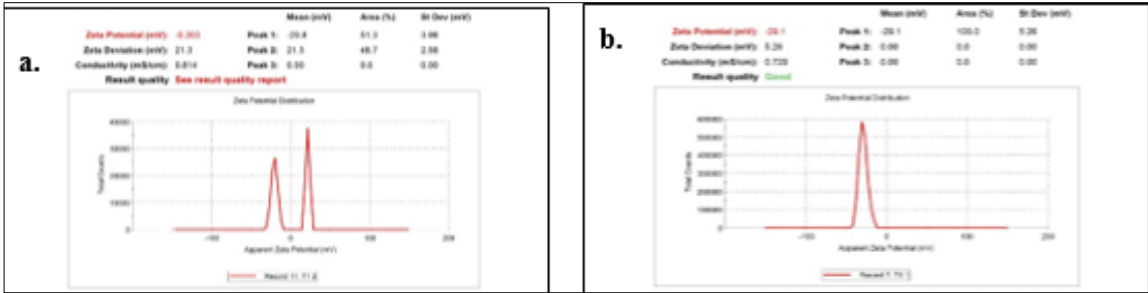


Fig 2: Zeta Potential of foam-mat dried jamun powder a) T1 (5%SPI, 2%CMC) b) T5 (1% SPI, 2%CMC)

3.3 Scanning electron microscopy-energy dispersive x-ray spectroscopy (sem-eds)

The scanning electron microscope (SEM) is a highly adaptable tool widely used for detailed investigation of surface morphology, microstructural features, and elemental composition of materials [12]. SEM imaging revealed clear morphological differences between the two formulations (Fig3) The high-SPI powder (T1) displayed a compact, aggregated structure composed of irregular, angular fragments in the 10-50 μm range with a rough surface texture, indicative of effective foam stabilisation and a well-reinforced particle network. The low-SPI powder (T5) instead showed a more porous, open structure with larger, rounded particles (20-80 μm) and greater void space, reflecting weaker structural reinforcement at the lower protein concentration. Despite these morphological differences, EDS elemental analysis showed both powders were dominated by carbon and oxygen at a broadly similar ratio (around 1.31:1 in T1

and slightly lower in T5), consistent with the organic composition expected of a fruit-derived powder.

3.4 FTIR spectroscopy

FTIR analysis in Fig 4a and Fig 4b showed that the high-SPI powder (T1) produced fewer, sharper and more clearly resolved absorption bands, indicative of strong protein-polymer interactions and a stable, well cross-linked matrix in which molecular breakdown during drying was minimised. The low-SPI powder (T5), in contrast, displayed a greater number of bands of varying intensity, suggesting weaker structural integrity and greater exposure of diverse functional groups originating from phenolics, carbohydrates and degradation product This pattern with sharper, simpler spectra at higher SPI concentration is consistent with findings by [8] who reported that higher SPI concentrations enhance matrix stability and reduce spectral complexity in foam-mat dried powders.

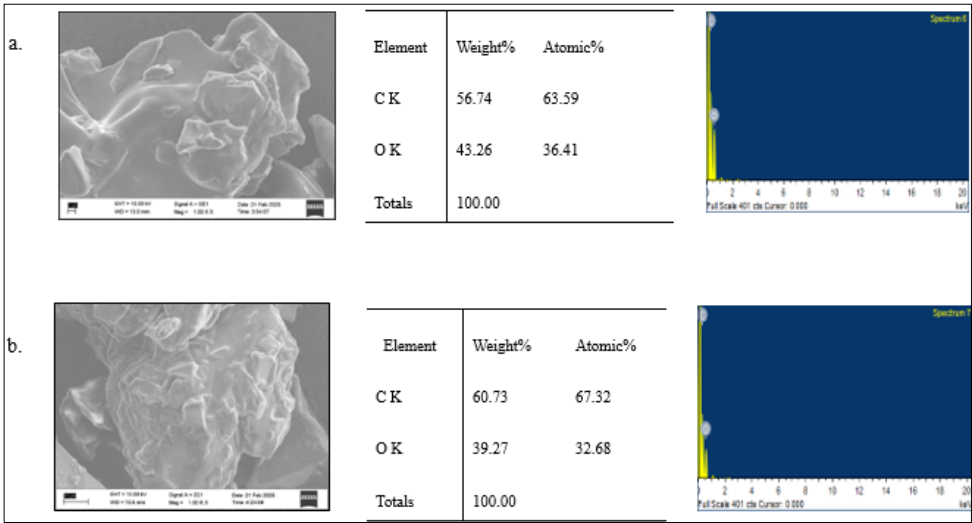


Fig 3: SEM-EDS spectrum images of foam-mat dried jamun powder a) T1(5%SPI, 2%CMC) b) T5 (1% SPI, 2%CMC)

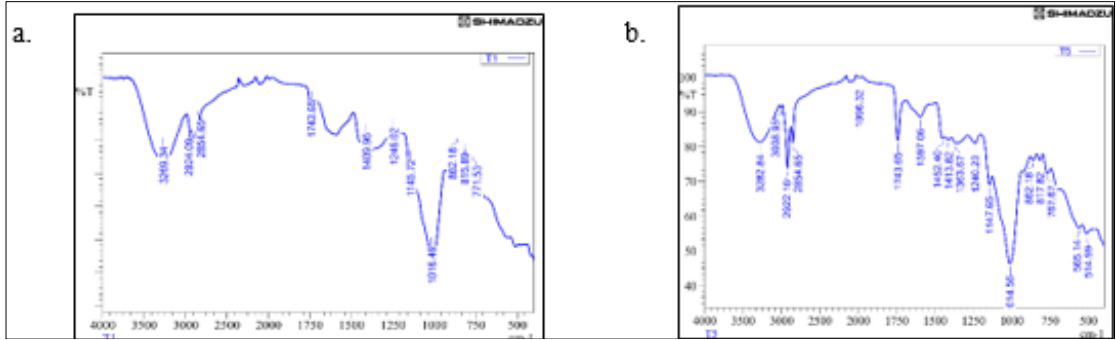


Fig 4: FTIR spectrum of foam-mat dried jamun powder a) T1(5%SPI and 2%CMC), b) T5 (1%SPI, 2%CMC)

3.5 UV-Visible spectroscopy

The UV-Vis analysis in Fig5a and fig5b showed that T₁ (5% SPI, 2% CMC) had higher absorbance (~4.0 in the UV region) and a distinct anthocyanin peak at 581 nm (Abs 0.43), indicating better retention of phenolics and pigments. The higher SPI content likely improved foam stability and

formed a protective matrix, minimizing anthocyanin degradation during drying. In contrast, T₅ (1% SPI, 2% CMC) showed lower absorbance (0.56 at 271 nm) and a weaker anthocyanin peak at 516 nm (Abs 0.20), suggesting reduced pigment retention due to insufficient foam structure and greater exposure to degradative conditions [11]

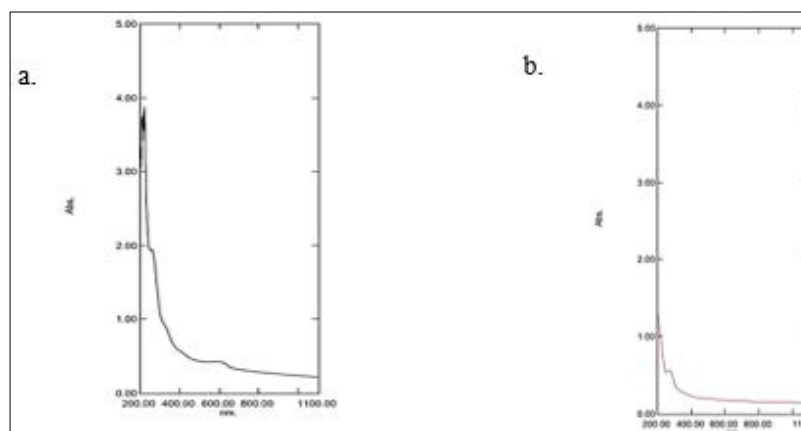


Fig 5: UV-VIS spectrum of foam-mat dried jamun powder a) T₁(5%SPI, 2%CMC) b) T₅ (1% SPI, 2%CMC)

4. Conclusion

Multi-technique Physico-chemical and structural characterisation showed that soya protein isolate concentration is the principal driver of particle architecture and molecular organisation in foam-mat dried jamun powder. At the higher SPI level (5% SPI, 2% CMC), the powder formed smaller, more uniform particles with a compact, angular SEM morphology and simpler, sharper FTIR and Raman spectra, reflecting a well cross-linked, structurally stable protein-polysaccharide matrix, but at the cost of a lower zeta potential magnitude. At the lower SPI level (1% SPI, 2% CMC), the powder formed larger, more heterogeneous particles with a porous, open SEM morphology and more complex FTIR and Raman spectra, indicating weaker matrix reinforcement, but with substantially greater electrostatic stability in dispersion. EDS confirmed that both formulations retained an organic, contamination-free composition dominated by carbon and oxygen. These findings demonstrate that SPI concentration can be used to tune the particle-scale structure and colloidal behaviour of foam-mat dried jamun powder, and that formulation choice should be guided by whether structural compactness or dispersion stability is the priority for the intended application.

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