Freeze-dried tomato powder optimization: pre-freezing temperature and physical forms impact on the physicochemical, structural, and functional properties

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Abstract
Controlling the freezing temperature is an essential stage in the freeze-dry process, in order to maintain the functional characteristics of food, such as viscosity, texture and physicochemical properties. Hence, this study assesses the impact of tomato shape (slice, 6% juice, 14% concentrate) and pre-freezing temperature (-24 °C vs. -80 °C) on the quality of freeze-dried tomatoes powder. Both factors significantly ($p<0.05$) affected the physicochemical, microstructural, and rheological properties. Slower freezing (-24 °C) improved lycopene retention in slices, decreased moisture in 6% juice, and lowered pH in 14% concentrate, but it also resulted in larger ice crystals and structural collapse. Faster freezing (-80 °C) produced finer micro-porosity and retained moisture. All reconstituted samples showed shear-thinning, according to rheological study; freezing at -24 °C produced increased viscosity and yield stress, indicating denser matrices. In general, both physical form and freezing rate affect product quality, with a trade-off between nutritional retention (preferred by little processing and faster freezing) and textural robustness (favored by slower freezing and greater solids).
Keywords: Freeze-drying, tomato powder, pre-freezing, Physical forms, Microstructure, Rheology.

Introduction

Drying is the most popular method of food preservation The water level of the food is lowered to a level where spoilage microorganisms cannot multiply or chemical reactions can occur. Beyond the preservation, drying extend the shelf life of dried foods [1]. Different drying technologies and types of equipment have been developed and their accuracy has increased over time. Sun, spray, microwave, osmosis, extraction, freezing can all be used for drying [2]. Freeze-drying (FD) is one of the most widely used methods for extending food shelf life. Freezing the water content of the material before freeze-drying prevents chemical, biochemical, and microbiological reactions [3, 4]. Tomatoes are an important agricultural crop and a major component of the global human diet [5]. Tomato (Lycopersicon esculentum) is a member of Solanaceae family.

Due to its balanced composition of nutrients and phytochemicals, tomatoes play an important role in human nutrition and health because they contain vitamin C, provitamin A (such as beta-carotene), vitamin E, minerals (such as potassium and magnesium), bioactive substances, and the pigment lycopene, a carotenoid that gives tomatoes their distinctive red color and antioxidant capacity [6]. Tomato powder is commonly used as a flavoring and coloring agent in food preparation [7]. Tomato powder has good potential as a substitute for tomato paste and other tomato product [8, 9].

However, due to high moisture content and water activity, tomatoes are susceptible to microbial growth and senescence, causing about 30% annual postharvest moisture loss, and freshly ripened tomato fruits deteriorate faster when stored in market or exposed to ambient conditions [10]. As a result, farmers are forced to sell their produce for less than they want. The price decline and loss of this lycopene-rich crop after harvest have been attributed to the lack of protective infrastructure from factors [11].

Reducing postharvest waste, improving product quality, and developing value-added tomato products that promote regional agriculture are all necessary. Therefore, the purpose of this study aims to optimize freeze drying, one method of preservation. with the goal of examining how freezing temperatures affect the physical quality of freeze-dried tomato products made in various forms (tomato slices, 6% tomato juice, and 14% tomato juice). In particular, the study examines the effects of freeze drying at two freezing temperatures (-24°C and -80°C) to ascertain their impact on tomato structural and nutritional integrity.

Materials and Methods

Sample preparation

Ripe, fresh tomatoes (Lycopersicon esculentum Mill.) were obtained at the local market of the Penjwen/ Iraq Kurdistan region. All the Tomatoes were sorted visually for color, size and physical damage. Divided into three portions. One portion, the tomatoes cut into cubic of 10 x 10 x10mm^3 [12]. Second one used to obtain tomatoes juice (6%), by homogenization, extraction blender - [tefal 8143, china], and filtration to remove seeds and peels Siever [prufsieb 180 µm]. The third portion, the tomatoes juice was concentrated by heating to (14%), after extract and filtered.

Freeze-drying process

Two different experiments were conducted, by testing two freezing temperatures, one at Ultra-Low Temperature Freezer -80°C, and the second at -24°C. The juice 6%, 14%, and the tomatoes slice were put in each 500ml bottle and freeze-dried by using a freeze dryer (Operon Ultra-Low Temperature Freezer -86°C, Korea) [13, 14] Average freeze-drying time was approximately 72 hours. All dried samples were milled and stored in desiccators at room temperature until further analysis.

Physic chemical evolution of row tomatoes and powdered

Fresh tomatoes and prepared freeze–dried powered tomatoes samples were determined by physic-chemical analysis such as moisture content, total solids content, total soluble solid (°Brix), acidity, and pH, according to [15].

Table (1): Physicochemical Properties with Lycopene cotenants mg/100gm of fresh tomatoes samples.

sample	Moisture %	Dry matter %	Acidity %	pH	Lycopene
slice	93.80 ±0.4 ^{A}	6.2 ± 0.2 ^{B}	0.24 ± 0.0 ^{C}	4.42±0.0^{C}	7.107± 0.1 ^{A}
Juice 6%	93.89±0.5 ^{A}	6.11 ± 0.0 ^{B}	0.27 ± 0.0 ^{B}	4.56 ± 0.0 ^{A}	2.442±0.2 ^{B}
Juice 14%	85.96 ±0.0 ^{B}	14.04±0.0 ^{A}	0.36 ± 0.0 ^{A}	4.48 ± 0.0 ^{B}	7.213±0.2 ^{A}

The mean± SD of three replicates is used to display the values. Within a same row, values with distinct superscripts show statistically significant differences ($p<0.05$).

Lycopene Content

Lycopene content was determined using titration method, according to [16]. Where 5 g of tomato puree was accurately weighed in a 150 ml beaker. 50 mL hexane-acetone ethanol solution (2:1 sample to dissolve lycopene was prepared and was allowed to stand for 5–10 min, then diluted in a 250 mL separatory funnel to separate the bilayers. Avoid light oxidation for spectrophotometric analysis. The acetone extract was then carefully mixed with 10 to 15 ml of petroleum ether in a separatory funnel. Petroleum ether was used to remove the pigment from the acetone extract. He went through this process several times until he was colorless. The pigment phase was transferred to a volumetric flask, and a spectrophotometer was used to measure the absorbance at 503 nm. the lycopene content was determined according to the equation1:

$$\text{Lycopene (mg/100g)} = \frac{A503 \times 31.2}{\text{Weight of sample gm}} \quad \text{………… (Eq.1(}$$

Microstructure

The scanning electron microscope (SEM) was used to measure the microstructure of frozen tomatoes through a tabletop scanning electron microscope, i.e. the MIRA3 TE SCAN was sprayed with a gold layer, and then placed in the measurement chamber [17]. The surface was observed at a magnification of 5,00 Kx and the cross-section at a magnification of 50. The system performs two main functions: data acquisition and image reconstruction.

Determination of Rheological property

A rheometer (MCR 302) was used to measure the rheological properties of the samples. Flow Behavior Analysis Shear stress and apparent viscosity were measured.20 g samples placed in a vial of the apparatus for rheological analysis. Every rheological test was carried out at temperature (25 °C) Each of the dry samples was made with tomato paste solution of 25% concentration according to the moisture content of the samples [18].

The Bingham model (Eq. 2) was used to analyze the flow behavior and viscosity of the reconstituted tomato concentrate:

$$\tau = \tau + \eta\dot{\gamma} \qquad \text{(Eq.2)}$$

Where: τ is the shear stress, $\dot{\gamma}$ is the shear rate, τ is the yield stress and η is the Bingham viscosity.

Statistical Analysis

Three replications using the Completely Randomized Design (CRD). The data collected were statistically evaluated using IBM SPSS (2021), version 27.0.1.0. The significance of the mean results at $P<0.05$ was assessed using a Duncan test.

Results and Discussion

The presented data in the (Table.2) shows the significant effect of tomatoes physical form (juice, concentrated juice 14%, and slicing) and two different freezing temperature (-24 °C vs, -80 °C) on the physicochemical properties of tomato products after freeze-drying process. The results show a significant difference ($p<0.05$) between the treatments. As moisture considered one of the important parameters during storage of dried material [19].

Hence, the comparison focused on the moisture content of the treatments. The 6% juice sample had the lowest moisture content when frozen at -24°C. This implies that the higher solids concentration due to this particular low-concentration 6% juice, and slower freezing mixture, was probably the result of synergy (removal of water from the solids). On the other hand, both 14% juice samples and frozen slices retained more moisture at −80°C. Rapid freezing at −80°C may result in smaller ice crystals that are better water retainers due to less structural damage to the cellular matrix [20].
Although the juice (14%) sample has lower moisture than 6% juice, the effect of freezing temperature is still noticeable. The juice at -80°C exhibiting a tendency toward somewhat higher moisture retention.

The data in the Table 2 also reveals a clear trend of the acidity and the pH of all the treatments. Juice 14% frozen at -24°C had highest acidity (0.39%) and lowest pH (4.21). this was contributing to the concentration process of the solids, which would also lead to increase the organic acids (e.g. citric acid and malic acid) in the tomatoes juice. The comparison between juice 14% frozen at -24°C and -80°C, the results showed that the freezing temperature also played a role. As for given sample, freezing at -80°C resulted in a slightly lower acidity and higher pH compared to -24°CThe warmer freezing temperature may cause varying degrees of chemical precipitation or slower, continuous enzymatic activity [21].

Table (2): Physicochemical analysis of Tomatoes samples after drying.

Sample	Moisture%	Dry matter %	Acidity %	pH
Slise-24°C	0.3^{C}±7.5	0,2^{B}±92.5	0.003±0.38 B	0.002±4.29 C
Slise-80°C	0.01± 8.01 AB	0.01±91.99 D	0.003±0.38 B	0.026 ±4.32 B
Juice%6-24 °C	0.002^{D}±6.5	0.02±93.43 A	0.0003±0.34 C	0.004±4.37 A
Juice%6-80°C	0.004^{B}±7.87	0.03±92.13 D	0.01±0.32 D	0.004±4.37 A
Juice%14-24°C	0.002±7.65 C	0.02±92.35 C	0.0002±0.39 A	0,01^{D}±4.21
Juice%14-80°C	0.01±8.16 A	0.02±91.84 E	0.004±0.38 B	0.023±4.27 C

The mean± SD of three replicates is used to display the values. Within a same row, values with distinct superscripts show statistically significant differences ($p<0.05$).

Lycopene

The physical characteristics of tomatoes and the impact of freezing temperatures on lycopene concentration (mg/100g) are shown in Figure 1. The highest lycopene content (31.125 mg/100g) was found in the Slice -24°C, indicating that there was no degradation at room temperature. This suggests that fresh slicing without heat treatment successfully preserves lycopene. In the meantime, there was a notable decrease (25.178 mg/100g) in the tomato slice samples that were frozen at an extremely high temperature (Slice-80°C). When compared to (Slice-24°C), the lycopene concentration dropped by about 19%. Additionally, Juice %6-24°C and Juice %6-80°C had lower lycopene levels (25.534 and 22.042 mg/100g, respectively) than their sliced equivalents. The mechanical disruption of juicing may expose lycopene to light and oxygen, hastening its degradation [22]. Additionally, heating juice exacerbates this loss [23]. Juice %14-24°C and Juice %14%-80°C had the lowest lycopene level (20.545 mg/100g), suggesting that greater concentrations or longer processing times may hasten lycopene breakdown.

Overall, the data points to a negative correlation between processing intensity and lycopene retention, particularly when heat and mechanical disturbance are present According to this study [24].

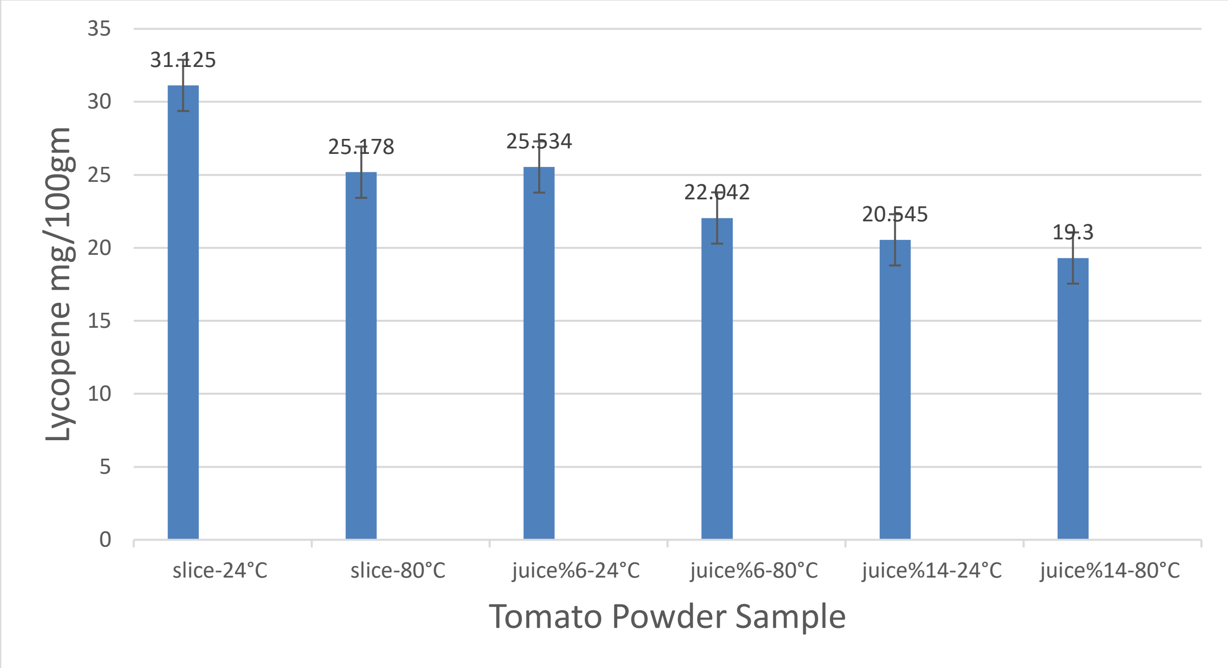


Figure (1): Lycopene content of freeze-dried tomatoes powder (6% juice,14% juice, and slice), freezing at 24 °C and -80 °C.

Microstructure and SEM

The topography and morphology of tomato powder samples were qualitatively evaluated using SEM. Figure 3 illustrates the effects of freezing temperature and physical shape (slice versus juice at 6% and 14% solids) on surface topology, matrix collapse, and ice-crystal damage.

The morphologies are compatible with conventional occurrences caused by freezing. Higher subzero temperatures result in larger crystals and significant breaking, as opposed to finer, denser formations at lower temperatures.

Large, smooth, sheet-like lamellae with broad spans and soft folds are seen in Figure 3-A (tomato slice freezing in -24°C). It is clear that delayed nucleation results in bigger ice crystals that tear and push parenchyma, generating macro-scale delamination and flattening cell walls into sheets [25]. Furthermore, Figure 3-B shows that freezing at -80°C results in a lot of microscopic fine porosity with less dramatic wall rupture; this finding agrees with [26].

According to [27], low-solids tomato juice (%6 freezing at -24°C) produces a weak biopolymer network in picture C. Larger crystals separate the gel at −24°C, causing lamellar stratification, creasing from ice segregation, and ultimately collapse. This feature's high porosity and layer delamination indicate cooperation and phase separation upon freezing; weak body and watery mouthfeel are likely. In contrast, picture D exhibits less lamellar failure and higher micro-scale uniformity than image C. Although it is still porous, the stability against macroscopic phase separation has increased. This is caused by serum traps and exquisitely carved crystals from quick solidification. According to [27], solute concentration has a substantial impact on the size and shape of ice crystals.

Furthermore, Image E illustrates the characteristic of long, fibrous threads with granular deposits on surfaces. Proteins, pectin, and insoluble fiber are higher solids that strengthen the network. Larger crystals stretch the matrix into strands at -24°C, while solute granulation on fiber surfaces is driven by freeze-concentration. On the other hand, image F displays a compacted microstructure with thick, highly textured folds.

Overall, at −24°C, big-crystal imprinting features (broad sheets, lamellae, large voids) are frequently observed, while at −80°C, denser textures, finer porosity, and micro-wrinkling are created. In addition, in respect to the shape effect (slice versus liquid): While slices have sheet-like wall remnants and intercellular delamination, juices exhibit gel-like stratification at low solids.

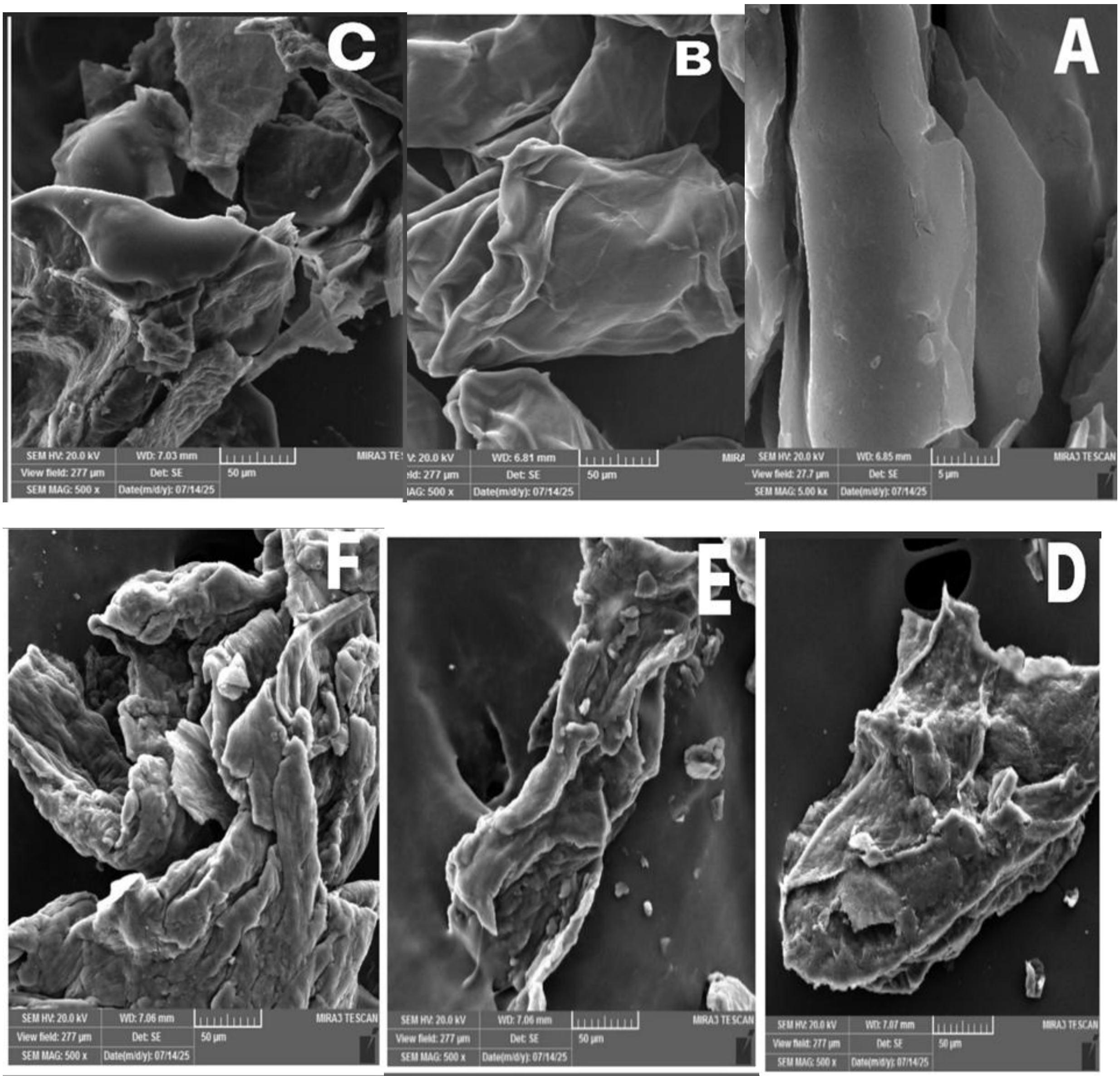


Figure (2): SEM images of tomatoes powder: A (tomato slice freezing in -24°C), B (tomato slice freezing in -80°C), C (tomato juice %6 freezing in -24°C), D (tomato juice %6 freezing in -80°C), E (tomato juice %14 freezing in -24°C), F (tomato juice %14 freezing in -80°C).

Rheological property

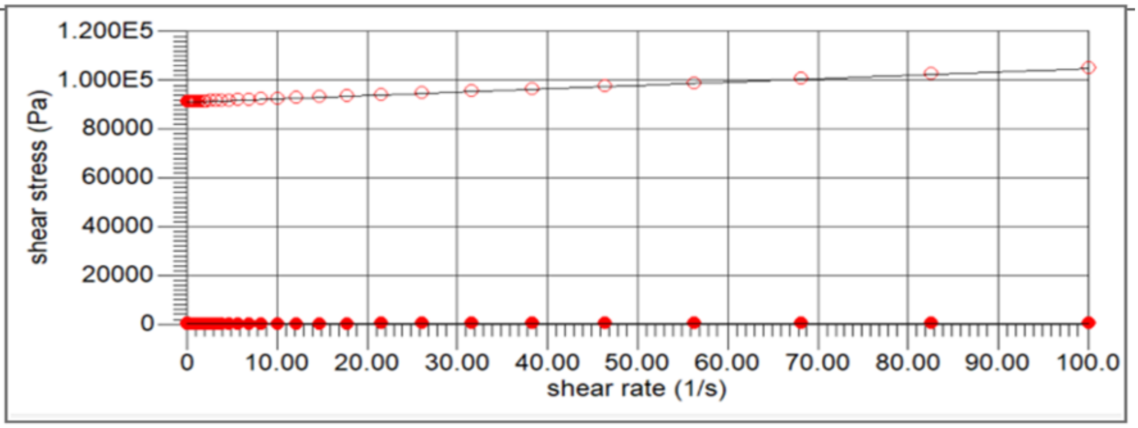
The rheological behavior of reconstituted tomato products under different shear rates at 25°C is shown in Figure 3, which also compares the impact of concentration levels (6% vs. 14%) and freeze-drying temperatures (-24°C vs. -80°C) in juice and slice forms. Shear stress rises with shear rate in all samples, displaying non-Newtonian, shear-thinning behavior. Tomato-based fluids show non-Newtonian, shear-thinning behavior because of their complex colloidal structure and polysaccharide content, such as pectin, as noted by [28].

Samples processed at -80°C consistently exhibit lower shear stress values than samples processed at -24°C in all panels (A–C). The best model to fit tomato paste flow curves was determined to be the Bingham model. According to [29], the yield stress considerably dropped with temperature and rose with concentration.

Reconstituted slices show higher shear stress values than juice samples under comparable freeze-drying conditions, according to Graph C. Slices probably hold onto more fibrous and insoluble components, which raises viscosity and yield stress. Additionally, uniform dispersion may be hampered by the structural complexity of slices, resulting in uneven flow behavior. This could be explained by remaining fibrous components in slices or insufficient solubilization, both of which increase apparent viscosity.

When Graphs A and B (6% juice vs. 14% juice) are compared, 6% juice shows higher viscosity and shear stress values, suggesting more structural rigidity and less uniform dispersion upon reconstitution. The 14% juice can be heated to hydrolyze pectic materials and other long-chain carbohydrate polymers, producing smaller molecules [28].

A



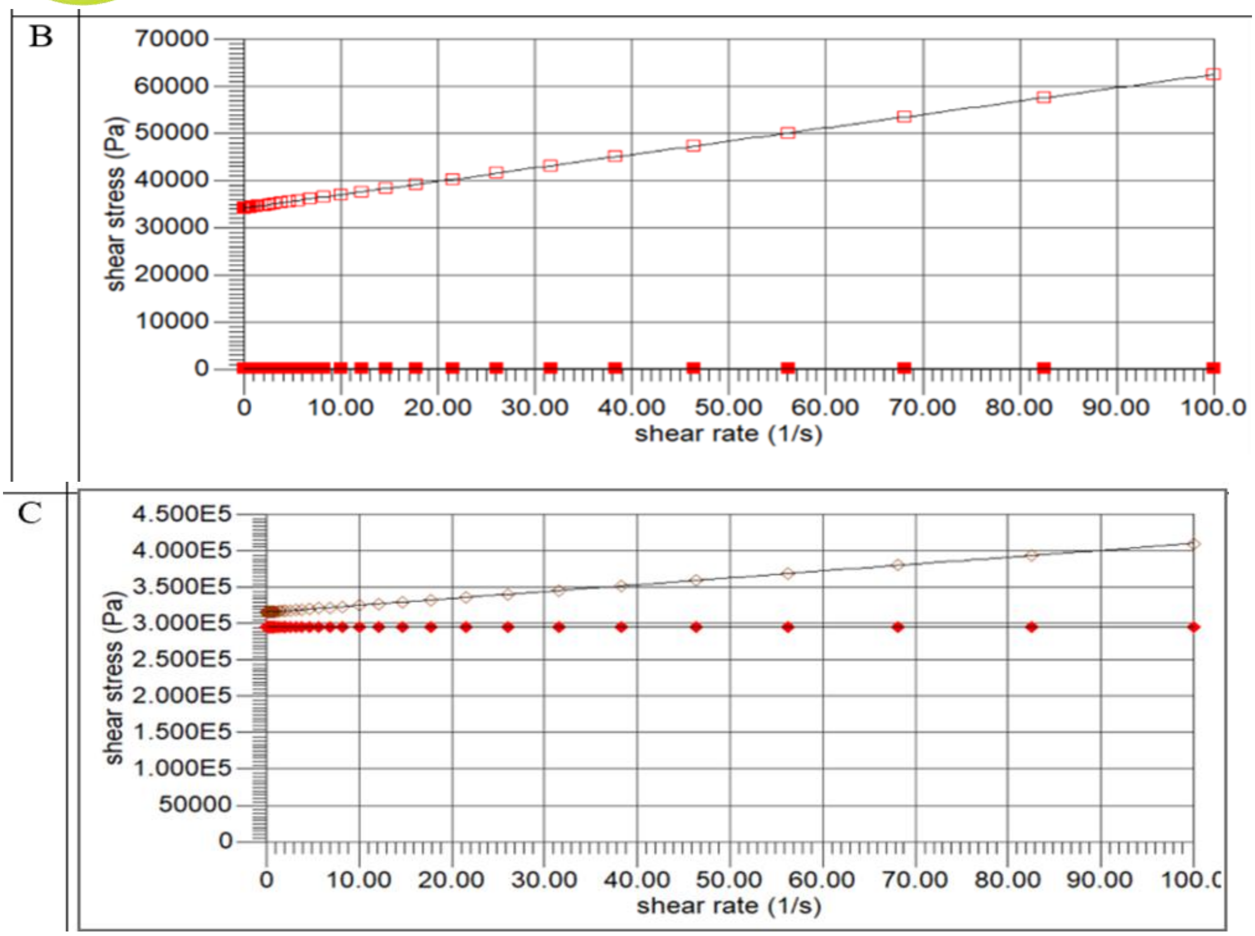


Figure (3): Flow behavior curves for reconstituted tomatoes powder at temperature 25 °C, fitted by Bingham model: A (hollow circle: 6% tomatoes juice -24 °C, and filled circle: juice 6%-80 °C), B (hollow square:14% tomatoes juice -24 °C, and filled square: 14% juice -80 °C), and C (hollow diamond: tomatoes slice -24°C,and filled diamond: tomatoes slice -80 °C).

Every tomato sample flow was fitted using the Bingham model. [29] state that tomato paste and reconstituted tomato solutions have Bingham plastic behavior, which makes this model appropriate for describing their flow characteristics.

The Bingham model parameters produce yield stress (τ_0) and viscosity (η) (Table 3), which represent the samples' flow resistance and structural integrity. In comparison to samples dried at -80°C, those dried at -24°C consistently exhibit higher yield stress and plastic viscosity, indicating stronger internal structure and increased resistance to early flow. This implies that denser, less porous matrices that impede rehydration and raise flow resistance could be produced by freeze-drying at -24°C.

In line with the data, samples frozen at -80°C show much lower yield stress and viscosity than samples frozen at -24°C. For example, the yield stress and viscosity of the 6% juice sample decrease from 3415 Pa to 47.93 Pa and 283 Pa·s to 0.421 Pa·s, respectively. All sample types, including 14% juice and slices, exhibit this pattern.

Smaller ice crystals are probably produced by freezing at -80°C, which reduces structural damage during freeze-drying. Nevertheless, this also results in rehydrated matrices that are less dense, which lowers flow resistance. On the other hand, freezing at -24°C may result in larger ice crystals, which would increase yield stress and viscosity by generating greater cellular disruption and denser rehydrated structures.

This is in accordance with research by [30], who showed that tomato tissue mechanical characteristics and rehydration kinetics are significantly impacted by microstructural integrity during freeze-drying. In a similar [18] highlighted that composition and processing circumstances, such as freezing and drying parameters, have an impact on rheological qualities.

Table (3): Bingham model parameters of freeze-dried tomatoes reconstituted (6% juice,14% juice, and slice), freezing at 24 °C and -80 °C.

Sample	τ_0 Pa	R^2	η Pa. s	R^2
Slice-24°C	0.01±9.81	0.998	0.02±946	0.998
Slice-80°C	0.02±2.954	0.998	0.02±0.018	0.999
Juice%6-24°C	0.01±3415	0.999	0.1 ±283	0.998
Juice%6-80°C	0.02±47.93	0.998	0.421 ± 0.1	0.999
Juice%14-24 °C	4155 ± 0.02	0.998	138.3 ± 0.01	0.999
Juice%14-80°C	0.01±74.14	0.999	0.366 ± 0.02	0.998

mean± SD of three replicates is used to display the values. Within a same row, values with distinct superscripts show statistically significant differences ($p<0.05$).

Each graph (A, B, and C) illustrates the link between shear stress and shear rate and how tomatoes react to increased shear (flow). The shear stress values are greater than the specimens for the same shear rate. This suggests that the samples are more viscous and less fluid [29]. All samples exhibit Bingham plastic fluid behavior, with the exception of the tomato juice sample, which exhibits Cason plastic fluid behavior at freezing point -80°C [29]. This suggests that non-Newtonian fluid -80 is observed at -80°C; samples with strong conductivity and low drag may exhibit non-Newtonian properties and yield stress. Make sure they are light. Shear stress is significantly reduced in the same shear rate range when viscosity decreases, and curves become flatter and more linear, suggesting less yield stress and flow resistance.

As a result, in samples of tomato slices and 14% -80°C tomato juice, the material primarily behaved as a weak shear fluid. The rheological characteristics of tomato products were not considerably affected by prefreezing temperature prior to freeze drying, as evidenced by the fact that those frozen at −24°C did not exhibit similar variations in yield stress, viscosity, and flow behavior as those frozen at −80°C.In tomato water, yield stress and viscosity both significantly decreased by 6% (from 98689 Pa to 88340 Pa and 393.0 Pa·s to 253.2 Pa.s, respectively), suggesting a considerable decrease in structural resistance to flow [31] .The viscosity of tomato pulp (14%) decreased from 264.0 Pa.s. to 203.8 Pa.s. and the yield pressure decreased from 39260 Pa.s. to 2.954E5 Pa.s. This suggests that quick freezing at −24°C preserved the cellular architecture and polysaccharide network, resulting in a more viscous and denser product. By freezing tomato slices at -24°C and -80°C, yield stress (2.954E5 Pa) was also normalized. The flow behavior index (n = 0.3860) decreased as a result, and viscosity increased from 0.2815 Pa.s. to 1.036 Pa.s. These findings imply that severe freezing causes non-Newtonian behavior while increasing structural stiffness because of the thicker matrix created during lifting. Samples of 6% tomato juice frozen at −80°C showed greater structural stability and more non-Newtonian behavior (n < 1) [32].

This study clearly shows that the ultimate quality of freeze-dried tomato powder is determined by the physical form of the tomato and the pre-freezing temperature, which presents a trade-off for producers. Minimal processing (slicing) and slower freezing at -24°C maximized the retention of the bioactive component lycopene. However, this combination also produced a dense, lamellar microstructure, leading to reconstituted products with higher viscosity and yield stress. Conversely, juicing and rapid freezing at -80°C significantly degraded lycopene but yielded a superior functional powder. The ultra-low temperature created a fine, porous microstructure that resulted in the lowest moisture content and a product with significantly lower viscosity and flow resistance upon reconstitution. As a result, the selection of processing parameters depends on the application. While choosing juicing and -80°C freezing promotes practical qualities like quick rehydration and low viscosity, choosing slicing and -24°C freezing puts nutritional quality first. These results offer a clear framework for modifying tomato powder production to achieve particular functional or nutritional objectives in the development of food products.

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