Scientific Research Publishing

Food and Nutrition Sciences, 2026, 17(7), 648-657
https://www.scirp.org/journal/fns
ISSN Online: 2157-9458
ISSN Print: 2157-944X

Observation of the Effects of Spray Drying and Freeze Drying on the Physical Structure of Catfish Gelatin Using SEM

Joshua L. Herring

Department of Food & Animal Sciences, Alabama A&M University, Normal, AL, USA
Email: josh.herring@aamu.edu

How to cite this paper: Herring, J.L. (2026) Observation of the Effects of Spray Drying and Freeze Drying on the Physical Structure of Catfish Gelatin Using SEM. *Food and Nutrition Sciences*, **17**, 648-657.
https://doi.org/10.4236/fns.2026.177042

Received: June 25, 2026
Accepted: July 27, 2026
Published: July 30, 2026

Abstract

Catfish production (*Ictalurus punctatus*) in the United States was annually above 250 thousand tons in 2003 but dropped to approximately 150 thousand tons in 2012 and in 2023 reached approximately 170 thousand tons annually. Mississippi, Alabama, Arkansas, and Texas account for approximately 96% of total catfish sales. Approximately 55% of the whole weight of catfish is considered a by-product with 98% of it being sold as offal for ~3 cents per pound. Of this, 25% is skin and frame which contains collagen. This collagen can be extracted through partial hydrolysis to yield edible gelatin that can be utilized in value-added food and cosmetic products. Thus, catfish by-products are of great interest in the catfish industry. The objective of this research was to extract gelatin from channel catfish skins and compare the structural impacts of subsequent dehydration methodologies for targeted food applications. Catfish skins were sourced from a regional commercial processing plant. Structural disintegration was achieved via acid hydrolysis to transition the skins into a gelatin solution. The solution was filtered sequentially through cheesecloth to remove macro-particulates and activated charcoal to remove micro-particles, residual fats, and off-odors. The purified yield was split into two equal treatments: one portion was freeze-dried (FD) and the other was spray-dried (SD). Structural characterization was performed via a JEOL 6390 LV Scanning Electron Microscope (SEM). SEM micrographs revealed distinct morphological divergences: the SD treatment formed dimpled globular spheres, whereas the FD treatment exhibited a porous, interconnected fibrous sheet network. These microstructural variations alter the texturizing, hydrating, and encapsulating properties of the gelatin, establishing a processing-driven mechanism to customize byproduct gelatin for specialized functional niches in food formulation.

Keywords

Catfish Gelatin, Spray Drying, Freeze Drying, Scanning Electron Microscopy, Hollow Globules, Fibrous Structure, Aquaculture By-Products

1. Introduction

Gelatin is a high molecular weight protein extracted from animal collagen by thermal denaturation or physical and chemical degradation and gelatin's versatility and functionality reflect the fact that it is the only food protein that undergoes a thermally reversible helix coil partial transition to resemble its original parent protein structure, collagen [1]. Collagen has a triple helix structure which forms fibers arranged in bundles which make up the connective tissue matrix. Acid and alkaline hydrolysis lead to mild degradation and the fibrous structure of collagen is broken down irreversibly because of the rupture of covalent bonds. Denaturation of soluble collagen caused by destruction of the triple helical structure of collagen produces one, two or three random chain gelatin molecules that give a solution of high viscosity, depending upon concentration [2].

Fish gelatin has gained importance in recent years because of the Bovine Spongiform Encephalopathy (BSE) issue in cows and markets that do not accept any pig related or cow based food products [3]. The manufacture of fish gelatin would also reduce processing waste material and result in value-added products. Fish gelatin can be extracted from the low value by products of the fish industry like fish skins, bones, and swim bladders by which utilization economic and waste management for the fish industry is greatly improved [4].

The physical dehydration method chosen to preserve gelatin critically determines its final structural geometry and commercial utility. Spray drying is an efficient technique that allows the instantaneous drying of solutions, suspensions, or emulsions by atomizing them into a hot gas stream [5]. Commercially, spray drying is highly advantageous for food and pharmaceutical applications because it is roughly 30 to 50 times less expensive than freeze drying [6]. Conversely, freeze drying (lyophilization) is favored for heat-labile or highly unstable materials where structural integrity and high product quality are paramount. Despite yielding premium-quality products, freeze drying remains an energy-intensive, prolonged batch process with operating costs typically triple those of standard thermal methods [7]. It can also sometimes induce partial protein degradation during the freezing stresses or generate uneven cakes instead of fine, uniform powders [8].

Although many proteins have been stabilized successfully by freeze-drying, this technique has some serious drawbacks: it is time- and energy-consuming and therefore expensive. Besides, it often leads to incomplete recovery of the intact protein, because of process-induced degradation (*i.e.*, during the freezing and drying phases) [8]. Moreover, as freeze-dried procedures usually generate cakes ra-

ther than powders, it is not the drying method of choice when microparticles with defined, narrow size distributions are the target [9]. The use of freeze-drying in food industries is therefore restricted to high value-added products such as coffee and tea, ingredients for ready-to-eat foods (vegetables, pasta, meat, fish, etc.) and several aromatic herbs.

To date, comparative research detailing the physical-chemical effects of these distinct drying strategies on fish gelatin microstructures remains limited. Therefore, the objective of this study was to compare the structural morphology and mechanical performance of gelatin extracted from channel catfish skins dried via spray drying and freeze drying using scanning electron microscopy (SEM) and texture profile analysis (TPA).

2. Materials & Methods

Catfish skins were obtained from a commercial catfish processor in Alabama, USA. Skins were cut into small pieces (~2 × 2 cm^2) and washed three times in D.I. water to remove residual meat and fat. Cleaned skins were treated with a degreasing solution (0.25% $NaHCO_3$ + 1% NaCl). Degreased skins were pretreated with 0.05 M Acetic acid and gelatin was extracted by placing the treated skins in a water bath at 55˚C for 3 hours. Gelatin was filtered with a Büchner funnel with #4 Whatman filter paper followed by filtration in a 44 × 600 charcoal filtration system (to remove odor, dark color and residual fat).

Drying techniques were performed on the gelatin solutions of different concentrations.

Spray drying (SD) entailed utilizing a Büchi Mini Spray Dryer (B-290, Büchi Labortechnik AG, Schweiz) with a standard 0.7 mm nozzle and nitrogen as an unreactive carrier gas. Inlet temperature of 100˚C with an aspiration rate of 95 and pump speed of 10 mL/min was used. Spray dried powder was rehydrated and three concentrations 1.67%, 3.34% and 6.67% of gelatin solution were made. Freeze Drying (FD) utilized a Genesys 35L Freeze Drier (VirTis, SP Industries, Gardiner, NY). The gelatin was cooled to 15˚C to induce gelling. Freeze drying utilized a stepwise recipe increasing in temperature (starting at −40˚C and ending at 30˚C) over a period of 18 hours.

Texture Profile Analysis (TPA) was conducted on 1.67%, 3.34% and 6.67% solutions of gelatin. They were solidified at 15˚C in a cylindrical mold (2.8 cm × 2.8 cm). TPA was performed in triplicate using Brookfield LFRA Texture Analyzer at 4 mm compression and 1mm/s probe speed. Peak load (hardness), gumminess, chewiness, springiness and percent deformation were measured.

Dried SD and FD gelatin specimens were mounted and sputter-coated with gold. Microstructural exploration of the surface and internal cross-sections was performed using a JEOL 6390 LV scanning electron microscope (JEOL, Tokyo, Japan). A minimum of five fields of view per sample were screened to select the most representative microphotographs.

Statistical analysis was conducted on data obtained from the texture profile

analysis (including peak load, gumminess, chewiness, springiness, and percent deformation) to determine the significance of differences across the three gelatin concentrations (1.67%, 3.34%, and 6.67%). A one-way Analysis of Variance (ANOVA) was performed to test for overall treatment effects. Following a significant main effect, post-hoc pairwise comparisons of the concentration means were executed using Student's t-test. Statistical significance was evaluated at an alpha level of $\alpha = 0.05$. All data processing and statistical computations were conducted using SAS software (Version 9.4; SAS Institute Inc., Cary, NC, USA).

Results & Discussion

Texture profile analysis highlighted a direct relationship between gelatin polymer concentration and gel network resilience. As shown in Table 1, increasing the gelatin concentration from 1.67% to 6.67% resulted in a substantial, greater-than-threefold increase in peak load (hardness), gumminess, and chewiness. Conversely, a 1.3-fold decrease in elasticity and deformation resistance was noted. These trends align closely with observations [10], confirming that higher concentrations accelerate peptide chain entanglement, thereby augmenting bloom strength and the force required to break the structural matrix.

Table 1. Texture profile analysis of 1.67%, 3.34% and 6.67% concentrations of gelatin samples.

PARAMETERS	1.67% GELATIN	3.34% GELATIN	6.67% GELATIN
PEAK LOAD (g)	$43.05^{a} \pm 0.5$	$86.33^{b} \pm 6.75$	$115.83^{c} \pm 8.04$
GUMMINESS (g)	$40.02^{a} \pm 0.27$	$73.46^{b} \pm 1.6$	$104.28^{c} \pm 3.32$
CHEWINESS (g.mm)	$128.67^{a} \pm 4.94$	$221.16^{b} \pm 11.95$	$311.97^{c} \pm 19.75$
SPRINGINESS	$3.24^{a} \pm 0.15$	$3.01^{a} \pm 0.13$	$2.99^{a} \pm 0.11$
% DEFORMATION	$14.27^{a} \pm 0.28$	$14.55^{a} \pm 0.15$	$14.59^{a} \pm 0.96$

^{abc} means in the same row with different superscripts are significantly different ($P < 0.05$).

Freeze dried products (Figures 1-6) maintained the matrix of the gel in a dried form. The structural elements of the gelatin matrix present a preferential spatial orientation due to ice crystallization [11]. If the product enters and exits the glass transition temperature, as the gel freezes, the water provides homogenous dispersion as a result of hydrogen bonding. It is believed that in a shorter freezing time the number of crystal nuclei formed is larger yet the nuclei will be smaller. Lv and Feng [12] discussed freeze drying methods as used to prepare three-dimensional fibroin scaffolds. Utilizing different fibroin concentrations (8% and 12%) they were able to produce scaffolds of interconnected pores of 100 μm in diameter with high yield strengths. With adjustments to the freeze drying technique, physical and mechanical properties of the gelatin could be altered to fit the desired application. Li and others [13] reported that silk fibroin scaffold porosity was below 70%, which was unacceptable for cell migration and expansion. Nazarov and oth-

ers [14] reported silk fibroin pore scaffold formation at about 50 μm with freeze drying. These results coupled with Lv and Feng [12] promote the use of freeze drying and a freeze drying/foaming technique as a means to create protein scaffolds which may be used in medical, pharmaceutical and food industries.

All SEM figures shown were chosen from a minimum of five samples for the most representative images from each treatment. **Figure 1** and **Figure 2** represent freeze dried structures from 1.67% freeze dried gelatin solution viewed at magnifications of 250X and 1700X, respectively. The pore structures were interconnected. Mostly open pore structures were observed. **Figure 3** and **Figure 4** represent 3.34% freeze dried gelatin solution at magnifications of 75X and 200X, respectively. These pore structures were not as interconnected as the 1.67% solution but contained mostly open pore structures. **Figure 5** and **Figure 6** represent 6.67% freeze dried gelatin solution at magnifications of 100X and 250X, respectively. The pore structures were not as interconnected as **Figures 1-4**. A mix of open and closed pore structures with closed pores dominating the matrix were observed at the higher concentration. Little homogeneity and consistency was found between samples in **Figures 1-6** in relation to pore circumference and area within the pore.

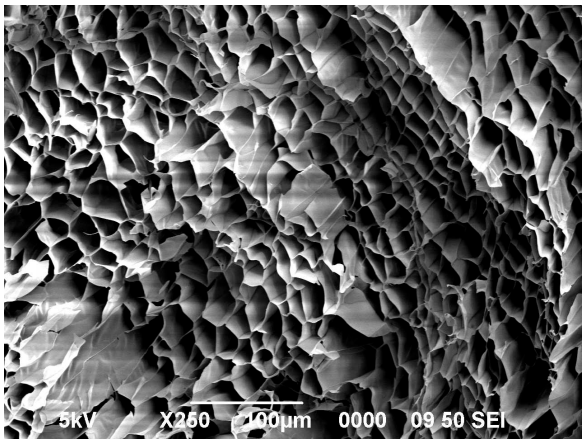

Figure 1. Freeze dried gelatin (1.67%) at magnification of 250X.

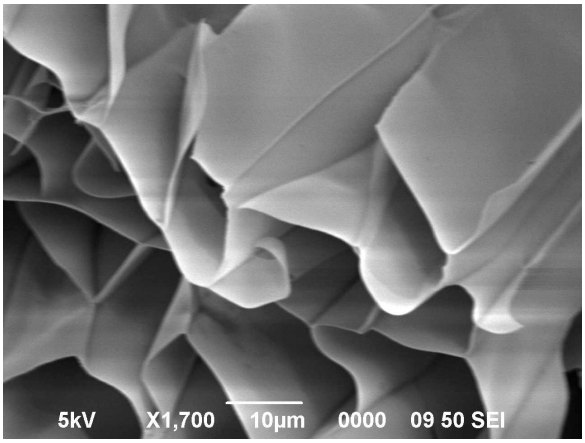

Figure 2. Freeze dried gelatin (1.67%) at magnification of 1700X.

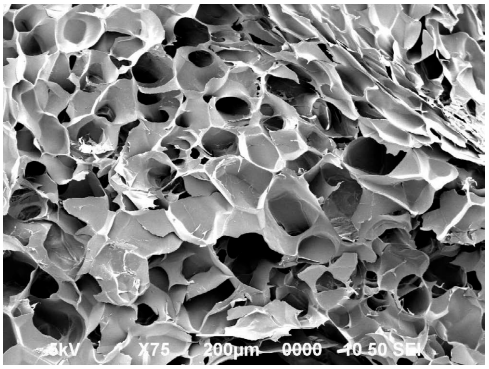

Figure 3. Freeze dried gelatin (3.34%) at magnification of 75X.

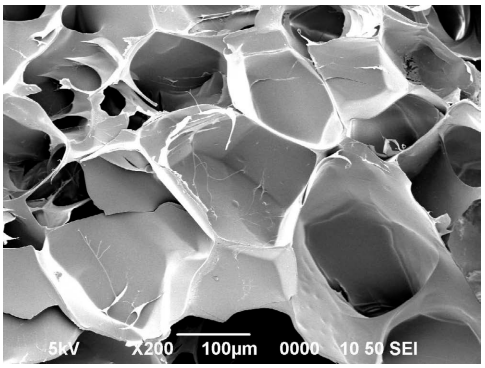

Figure 4. Freeze dried gelatin (3.34%) at magnification of 200X.

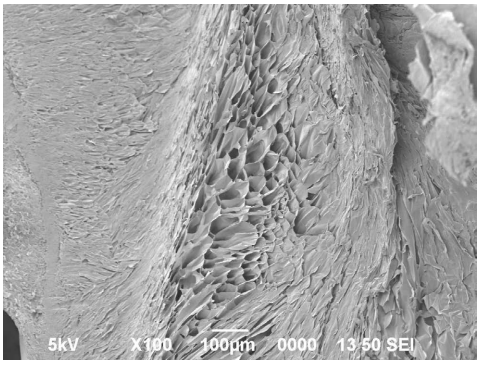

Figure 5. Freeze dried gelatin (6.67%) at magnification of 100X.

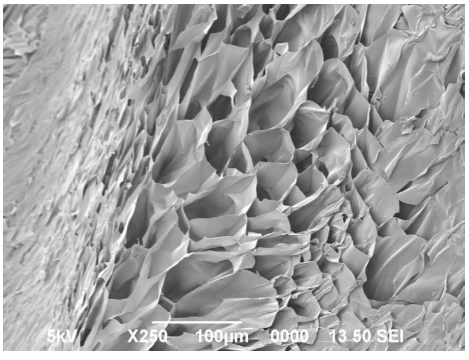

Figure 6. Freeze dried gelatin (6.67%) at magnification of 250X.

Spray dried product (Figures 7-12) was dried at 100˚C, 95% aspiration and pump rate of 10 ml/min. Figure 7 and Figure 8 represent the 1.67% spray dried gelatin solution at a magnification of 500X and 1500X, respectively. Figure 9 and Figure 10 represent 3.34% spray dried gelatin solution at a magnification of 500X and 1500X. Figure 11 and Figure 12 represent the 6.67% spray dried gelatin solution at a magnification of 50X and 500X, respectively.

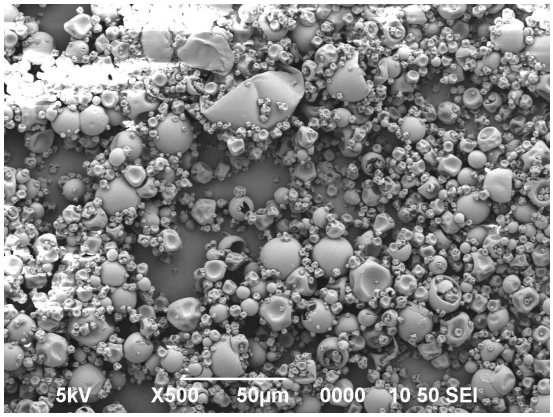

Figure 7. Spray dried gelatin (1.67%) at magnification of 500X.

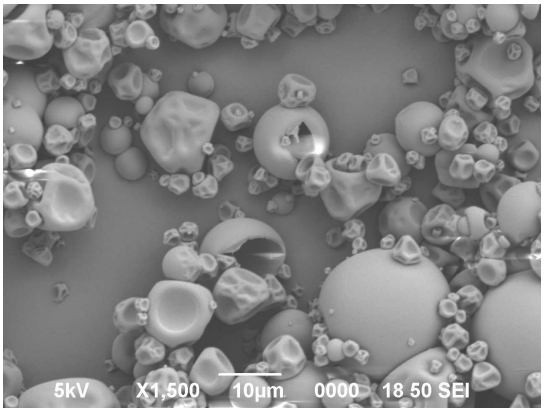

Figure 8. Spray dried gelatin (1.67%) at magnification of 1500X.

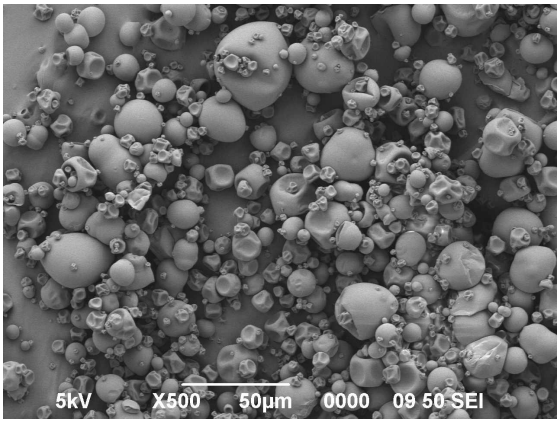

Figure 9. Spray dried gelatin (3.34%) at magnification of 500X.

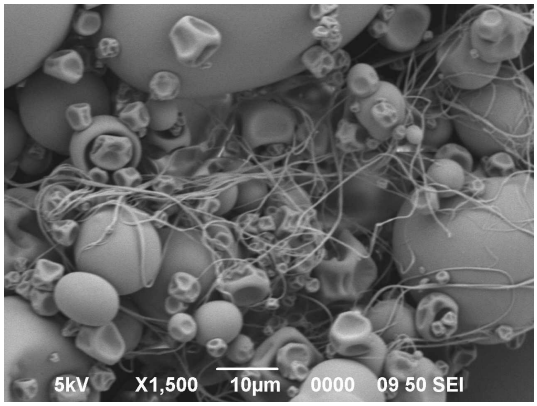

Figure 10. Spray dried gelatin (3.34%) at magnification of 1500X.

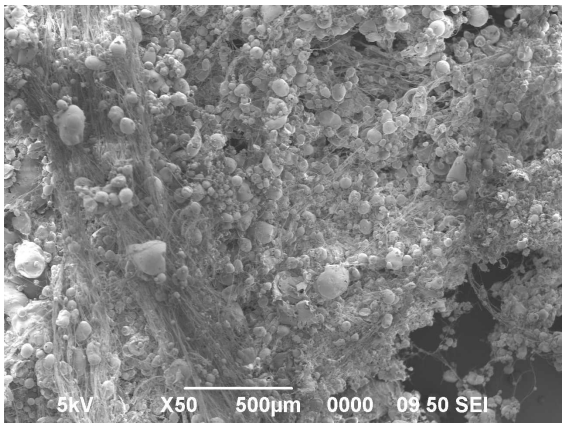

Figure 11. Spray dried gelatin (6.67%) at magnification of 50X.

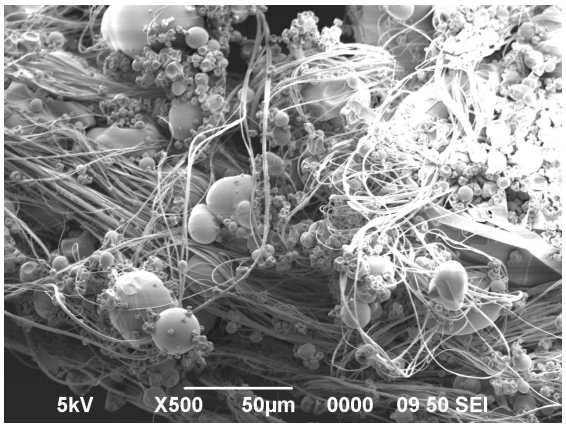

Figure 12. Spray dried gelatin (6.67%) at magnification of 500X.

Spivak [15] mentioned in a patent that hollow fibers are useful in reinforcing polymers and can be prepared by spray drying solutions of film-forming substances. Depending on the process settings, Spivak noticed that instead of hollow spheres formed via spray drying, hollow fibers were formed. Woo [16] reported that spherical globules of controlled size are generally produced by spray drying and that the granule structure can be optimized by adjusting the slurry parameters

and spray drying parameters. Kara and others [17] noted that the structural stability of the droplet and hydrodynamic effects during the spray drying process have important effects on particle morphology. The product that was achieved after spray drying had globules with shapes ranging from spherical to ellipsoid share with and without dimples/impressions.

3. Conclusions

The architectural divergence between freeze-dried fibrous matrices and spray-dried hollow or dimpled globules represents a powerful tool for tailored food formulation and structural engineering [5]. Rather than viewing these morphological anomalies as accidental process defects, modern food developers can exploit them to dictate texture, ingredient delivery, and reconstitution physics. The product development for catfish gelatin inclusion as an ingredient would allow producers and processors to have by-products as a revenue stream. The potential revenue could help offset the decrease in the catfish industry from approximately 250 thousand tons in 2003 to approximately 150 thousand tons in 2012 as it has not rebounded past approximately 170 thousand tons in 2023 [18].

For lower concentrated solutions of catfish gelatin, highly porous freeze dried products were obtained and the porosity decreased as the gelatin concentration in the solution increased. Spray drying lower concentrated gelatin solutions yielded a globular product which is highly desirable for uniformity in structure and could be employed for encapsulation. Spray drying of highly concentrated gelatin solution yields fibrous protein structures linked with globules, which is undesirable due to the reduced uniformity in the product and potential for increased hydration issues. Studies need to be made on the effect of parameters such as pump speed, aspiration and temperature on spray dried protein structure and size as spray dried products appeared hydrophobic when rehydration was attempted. Future research could evaluate the amino acids on the dried gelatin surface structure as the native form of gelatin is highly hydrophilic.

Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

References

[1] Stainsby, G. (1977) The Physical Chemistry of Gelatin in Solution. In: Ward, A. and Courts, A., Eds., *The Science and Technology of Gelatin*, Academic Press, 179-208.

[2] Flory, P.J. and Weaver, E.S. (1960) Helix [UNK] Coil Transitions in Dilute Aqueous Collagen Solutions. *Journal of the American Chemical Society*, **82**, 4518-4525. https://doi.org/10.1021/ja01502a018

[3] Pranoto, Y., Lee, C.M. and Park, H.J. (2007) Characterizations of Fish Gelatin Films Added with Gellan and K-Carrageenan. *LWT—Food Science and Technology*, **40**, 766-774. https://doi.org/10.1016/j.lwt.2006.04.005

[4] Choi, S.S. and Regenstein, J.M. (2000) Physicochemical and Sensory Characteristics of Fish Gelatin. *Journal of Food Science*, **65**, 194-199.

https://doi.org/10.1111/j.1365-2621.2000.tb15978.x

[5] Shishir, M.R.I. and Chen, W. (2017) Trends of Spray Drying: A Critical Review on Drying of Fruit and Vegetable Juices. *Trends in Food Science & Technology*, **65**, 49-67. https://doi.org/10.1016/j.tifs.2017.05.006

[6] Desobry, S.A., Netto, F.M. and Labuza, T.P. (1997) Comparison of Spray-Drying, Drum-Drying and Freeze-Drying for β-Carotene Encapsulation and Preservation. *Journal of Food Science*, **62**, 1158-1162. https://doi.org/10.1111/j.1365-2621.1997.tb12235.x

[7] Snowman, J.W. (1997) Freeze Dryers. In: Baker, G.G.C., Ed., *Industrial Drying of Foods*, Springer, 134-155. https://doi.org/10.1007/978-1-4613-1123-2_7

[8] Pikal, M.J. (1990) Freeze-Drying of Proteins. Part I: Process Design. *BioPharm*, **3**, 18-27.

[9] Gupta, R.B. and Chattopadhyay, P. (2002) Method of Forming Nanoparticles and Microparticles of Controllable Size Using Supercritical Fluids and Ultrasound (U.S. Patent No. 20020000681). U.S. Patent and Trademark Office.

[10] Montero, P. and Gómez-Guillén, M.C. (2000) Extracting Conditions for Megrim (*Lepidorhombus boscii*) Skin Collagen Affect Functional Properties of the Resulting Gelatin. *Journal of Food Science*, **65**, 434-438. https://doi.org/10.1111/j.1365-2621.2000.tb16022.x

[11] Shen, F., Cui, Y., Yang, L., Yao, K., Dong, X., Jia, W., *et al.* (2000) A Study on the Fabrication of Porous Chitosan/Gelatin Network Scaffold for Tissue Engineering. *Polymer International*, **49**, 1596-1599. https://doi.org/10.1002/1097-0126(200012)49:12<1596::aid-pi553>3.0.co;2-s

[12] Lv, Q. and Feng, Q. (2006) Preparation of 3-D Regenerated Fibroin Scaffolds with Freeze Drying Method and Freeze Drying/Foaming Technique. *Journal of Materials Science: Materials in Medicine*, **17**, 1349-1356. https://doi.org/10.1007/s10856-006-0610-z

[13] Li, M., Wu, Z., Zhang, C., Lu, S., Yan, H., Huang, D., *et al.* (2001) Study on Porous Silk Fibroin Materials. II. Preparation and Characteristics of Spongy Porous Silk Fibroin Materials. *Journal of Applied Polymer Science*, **79**, 2192-2199. https://doi.org/10.1002/1097-4628(20010321)79:12<2192::aid-app1027>3.0.co;2-0

[14] Nazarov, R., Jin, H. and Kaplan, D.L. (2004) Porous 3-D Scaffolds from Regenerated Silk Fibroin. *Biomacromolecules*, **5**, 718-726. https://doi.org/10.1021/bm034327e

[15] Spivak, M.A. (1982) Spray Drying Method of Preparing Hollow Fibers (U.S. Patent No. 4,409,158). U.S. Patent and Trademark Office.

[16] Homayoonfal, M., Malekjani, N., Baeghbali, V., Ansarifar, E., Hedayati, S. and Jafari, S.M. (2022) Optimization of Spray Drying Process Parameters for the Food Bioactive Ingredients. *Critical Reviews in Food Science and Nutrition*, **64**, 5631-5671. https://doi.org/10.1080/10408398.2022.2156976

[17] Iskandar, F., Gradon, L. and Okuyama, K. (2003) Control of the Morphology of Nanostructured Particles Prepared by the Spray Drying of a Nanoparticle Sol. *Journal of Colloid and Interface Science*,**265**, 296-303. https://doi.org/10.1016/s0021-9797(03)00519-8

[18] USDA (2024) 2023 Census of Aquaculture. National Agricultural Statistics Service. https://www.nass.usda.gov/Publications/AgCensus/2022/Online_Resources/Aquaculture/Aqua.pdf