### APPENDIX-A

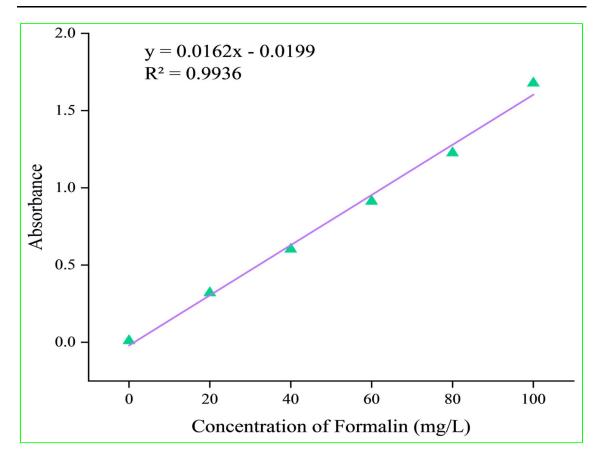


Fig. A1 Standard Curve of Acetylacetone method response to different formalin concentrations

### **APPENDIX-B**

### Publications, Participations and Recognitions from the Outcomes of the Research

### **B1 List of Publications**

- 1. **Dhua, S.**, & Mishra, P. (2025). Microwave drying: A novel technique in the sustainable development of corn starch-based aerogel and its comparison with traditional freeze dried aerogel. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 137135.
- 2. **Dhua, S.**, & Mishra, P. (2023). Development of highly reusable, mechanically stable corn starch-based aerogel using glycerol for potential application in the storage of fresh spinach leaves. *International Journal of Biological Macromolecules*, 242, 125102.
- 3. **Dhua, S.**, MJ, P. D., & Mishra, P. (2023). A comprehensive review on multifunctional smart carbon dots (C dots) based aerogel. *Food Chemistry Advances*, *3*, 100341.
- 4. **Dhua, S.**, Gupta, A. K., & Mishra, P. (2022). Aerogel: Functional emerging material for potential application in food: A review. *Food and Bioprocess Technology*, 15(11), 2396-2421.

### **B2** Journal articles under preparation

- 1. **Dhua**, S., Dulait, K., & Mishra, P. Freezing-assisted Microwave dried aerogel: An Innovative Method for Development of Aerogel.
- 2. **Dhua, S.**, & Mishra, P. Carbon Dots Loaded Starch-Based Functional Aerogel for determination of formalin content in Fresh Water Fish.

### **B3** Book chapters under preparation

1. **Dhua, S.**, Dulait, K., Bayan, A., & Mishra, P. Aerogel: an emerging functional material for food quality and safety analysis.

### **B4** Participation in National/International Conference

- **1. Dhua. S.**, & Mishra, P. Effect of glycerol on physical, mechanical, and morphological properties of corn starch-based aerogel and cryogel and its application in Food (Oral presentation) (ETSAFe- 2025) on 13<sup>th</sup> 15<sup>th</sup> February, 2025 organized by the Department of Food Engineering and Technology, Tezpur University, Assam, India in association with AFST (I) Tezpur Chapter.
- 2. **Dhua, S.**, & Mishra, P. Effect of humectant (glycerol) on physical and mechanical properties of corn starch based aerogel (Oral presentation) (Virtual poster presentation).

International Conference on Innovative Food System Transformations for Sustainable Development in Agro-Food and Nutrition Sector, organized by Department of Food Technology, Vignan's Foundation for Science, Technology & Research (Deemed to be University), Faculty of Agro-based Industry, Universiti Malaysia Lelantan (UMK), and Assoc. of Food Scientists & Technologists, CSIR- CFTRI Camus, Mysuru held online during 16th- 17th November 2022.

3. Dhua. S., & Mishra, P. Effect of humectant (glycerol) on morphology, functional properties of corn starch based aerogel (Oral presentation) (SAFETy- 2022) on 19<sup>th</sup> – 20<sup>th</sup> October, 2022 organized by the Department of Food Engineering and Technology, Tezpur University, Assam, India and Department of Soils, Water & Agricultural Engineering, Sultan Qaboos University, Oman in association with AFST (I) Tezpur Chapter.

### B5 Publications other than Ph.D.

- 1. Koch, P., **Dhua, S.**, & Mishra, P. (2024). Critical review on Citrus essential oil extracted from processing waste-based nanoemulsion: preparation, characterization, and emerging food application. *Journal of Essential Oil Research*, 36(5), 407-425.
- Yumnam, M., Gopalakrishnan, K., Dhua, S., Srivastava, Y., & Mishra, P. (2024). A
  Comprehensive Review on Smartphone-Based Sensor for Fish Spoilage Analysis:
  Applications and Limitations. Food and Bioprocess Technology, 17(12), 4575-4597.
- Gupta, A. K., Dhua, S., Kumar, V., Naik, B., Magwaza, L. S., Ncama, K., Opara U. L., McClements, D. J., & Mishra, P. (2023). Current and emerging applications in detection and removal of bitter compounds in citrus fruit juice: A critical review. *Food Bioscience*, 55, 102995.
- 4. Gupta, A. K., **Dhua, S.**, Sahu, P. P., Abate, G., Mishra, P., & Mastinu, A. (2021). Variation in phytochemical, antioxidant and volatile composition of pomelo fruit (citrus grandis (L.) osbeck) during seasonal growth and development. *Plants*, *10*(9), 1941.
- 5. Medhi, M., Gupta, A.K., **Dhua, S.**, Mishra, P. (2022). Food Additives. In: Chauhan, O.P. (eds) Advances in Food Chemistry. Springer, Singapore.
- Gupta, A. K., Dhua, S., Thakur, R., Ncama, K., Sithole, N. J., Magwaza, L. S., Naik, B., Mishra, P. (2023). Orange. In Fruits and their roles in nutraceuticals and functional foods (pp. 250–278). Boca Raton: CRC Press.

### **B6** Awards

1. Awarded **Third Prize** for the oral presentation on "Effect of glycerol on physical, mechanical, and morphological properties of corn starch based aerogel and cryogel and its

- application in Food" at ETSAFe- 2025 on 13th 15th February, 2025 organized by the Department of Food Engineering and Technology, Tezpur University, Assam, India in association with AFST (I) Tezpur Chapter.
- 2. Awarded **Best Poster Prize** for the poster presentation on "Effect of humectant (glycerol) on physical and mechanical properties of corn starch based aerogel". International Conference on Innovative Food System Transformations for Sustainable Development in Agro-Food and Nutrition Sector", organized by Department of Food Technology, Vignan's Foundation for Science, Technology & Research (Deemed to be University), Faculty of Agro-based Industry, Universiti Malaysia Lelantan (UMK), and Assoc. of Food Scientists & Technologists, CSIR- CFTRI Camus, Mysuru held online during 16th- 17th November 2022.



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### Colloids and Surfaces A: Physicochemical and Engineering Aspects

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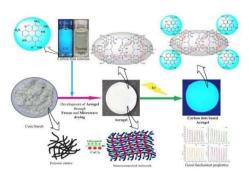
Microwave drying: A novel technique in the sustainable development of corn starch-based aerogel and its comparison with traditional freeze dried aerogel

Subhamoy Dhua, Poonam Mishra \*\*

I Sommi Principal

Department of Food Engineering and Technology, Tezpur University, Assam 784028, India

### GRAPHICAL ABSTRACT



### ARTICLEINFO

Keywords:
Corn starch aerogel
Microwave drying
Freeze drying
Freeze drying
Hysico-functional properties
Carbon dots (CDs) loaded aerogel

### ABSTRACT

A new way of developing corn starch aerogel through microwave drying has been investigated and the results have compared with the traditional corn starch aerogel developed through freeze drying. The effect of glycerol on both microwave and freeze-dried aerogel has also been investigated. Microwave dried aerogel possesses higher total shrinkage (76.09–87.00 %), density (0.33 – 0.76 kg/m²), and lesser water absorption capacity (144.03 – 213.75 %), porosity (49.02 – 77.95 %) as compared to freeze dried aerogel. However, microwave dried aerogel showed higher stability to thermal degradation and mechanical compression than freeze dried aerogel. The microwave dried aerogel showed good recompressibility as well as good reusability as compared to freeze dried aerogel. Less time taking process (28 min), ease of availability and operation, and less power consumption, etc. are the most important advantages of microwave dried aerogel development over freeze dried aerogel. Nitrogen doped Carbon dots (N-CDs) were grafted successfully into microwave dried aerogel. N-CDs loaded aerogel showed blue fluorescence when observed under UV light (365 mm). N-CDs loaded aerogel paves the way of microwave dried aerogel to be potentially applied in the purpose of sensing. It can also be employed as a functional material of different purpose (carrier matrix, moisture scavenger, etc.).

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<sup>\*</sup> Corresponding author.

E-mail address: mpoonam1@rediffmail.com (P. Mishra).



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### International Journal of Biological Macromolecules





Development of highly reusable, mechanically stable corn starch-based aerogel using glycerol for potential application in the storage of fresh spinach leaves

Subhamoy Dhua, Poonam Mishra

Department of Food Engineering and Technology, Tezpur University, Assam 784028, India

### ARTICLEINFO

Keywords:
Corn starch aerogel
Glycerol
Supercritical CO<sub>2</sub> drying
Physico-functional properties
Rehydration characteristics



Impact of glycerol on the physico-functional, morphological, mechanical, and rehydration properties of corn starch-based aerogel has been investigated. The aerogel was prepared from hydrogel (sol-gel method) using solvent exchange and supercritical CO<sub>2</sub> drying. Glycerol-infused aerogel had a more connected, denser structure (0.38–0.45 g/cm³), enhanced hygroscopic behavior, and was reusable up to eight times in terms of its capacity to absorb water after being drawn from the soaked sample. However, the inclusion of glycerol reduced the aerogel's porosity (75.89–69.91 %) and water absorption rate (WAR; 118.53–84.64 %) but enhanced its percentage shrinkage (75.03–77.99 %) and compressive strength (26.01–295.06 N). The most effective models for describing the rehydration behavior of aerogel were determined to be the Page, Weibull, and Modified Peleg models. Glycerol addition improved the internal strength of the aerogel so could be recycled without significant change in the physical characteristics of the aerogel. By effectively eliminating the condensed moisture that was developed inside the packing owing to the transpiration of fresh spinach leaves, the aerogel extended the storage life of the leaves by up to eight days. The glycerol-based aerogel has the potential to be employed as a carrier matrix for various chemicals and a moisture scavenger.

### 1. Introduction

International Union of Pure and Applied Chemistry (IUPAC) has defined aerogel as "non-fluid networks composed of interconnected colloidal particles as a dispersed phase in a gas (typically air)" [1]. Aerogel is a light, porous solid that has good mass transfer capabilities, a high specific surface area, low density, and high porosity. However, aerogel's definition is still ambiguous as its physicochemical properties are rigidly dependent upon the fabrication process (particularly on the drying process) [2]. Steven Kistler developed the first aerogel in 1931. Subsequently, he developed number of aerogels using various precursor materials (stannic oxide, silica, and cellulose). Despite the fact that aerogel was created about nine decades ago, its use only recently began [3,4].

Polysaccharides, proteins, and other bio-based precursor materials were uncommon in the past for the development of aerogel, but they are now relatively prevalent because of their unique characteristics, such as less toxic, eco-friendly, and biodegradable [3]. Aerogel produced from bio-based precursor materials is utilized for a variety of purposes,

including oil-water separation [5], thermal insulator [6], air filtration [7], ion exchange [8], food packaging [9–14], food and drug delivery systems [15–18], etc.

To produce aerogel, starch is a popular bio-based precursor material. Different kinds of starches were used to produce aerogel with a range of functional properties, including those for use as a bioactive filler and carrier in food preparations [19], a functional food ingredient [20], a chemical and bioactive component carrier [21–23], etc. The two main methods for creating hydrogel are sol-gel (aerogel monoliths) [10] and emulsion gelation (aerogel microspheres) [21]. Techniques like supercritical (SC) fluid drying and freeze-drying are frequently employed to convert hydrogel into aerogel. Supercritical drying is superior to freeze drying because it maintains the porous structure and generates pores in the nano range. [3].

In recent years, corn starch aerogel has gained interest due to its diverse application potential, especially in the food sector. In earlier research, SC-CO $_2$  drying (pressure: 80–200 bar, temperature: 37–45 °C) was used to create aerogel, with a starch concentration ranging from 7 to 15 % [2,6,10,20–22,24–30]. A high depressurization rate during SC-CO $_2$ 

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<sup>\*</sup> Corresponding author.

E-mail address; poonam@tezu.ernet.in (P. Mishra).



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### **Food Chemistry Advances**

journal homepage: www.elsevier.com/locate/focha



### A comprehensive review on multifunctional smart carbon dots (C dots) based aerogel



Subhamoy Dhua, Priya Darshini M J, Poonam Mishra\*

Department of Food Engineering and Technology, Tezpur University, Assam 784028, India

### ARTICLE INFO

Keywords: C dots Aerogel Luminescent behavior Sensing

### ABSTRACT

Till now from the time of invention of carbon dots (CDs), continuously grabbing the attention of the researchers due to their unique characteristics like nano size range (<20 nm), surface activity, photo luminescent behavior, non-toxicity, etc. Since, last decades CDs grab attention in the field of functional aerogel preparation. Functional CDs based aerogel is an emerging material which combines the characteristics of aerogel (high specific surface area, light weight, high porosity, etc.) as well as the CDs. Therefore, this review has been conducted to represent the current scenario of CDs based aerogel, their characteristics, and applications in various fields. The CDs based aerogel luminescent behavior at different wavelength, fluorescence quantum yield, and fluorescent quenching capability have found huge application in the field of sensing. Moreover, CDs based aerogel application in food sensing purpose is limited. Bio-based CDs aerogel with similar functional properties is an emerging area where further research is needed.

### 1. Introduction

According to IUPAC (International Union of Pure and Applied Chemistry), "aerogel are non-fluid networks composed of interconnected coloidal particles as dispersed phase in a gas (typically air)" (White et al., 2014). Almost nine decades ago, aerogel comes into use however, its application was limited in ancient time. Recently, aerogel have gained huge interest from the researcher due to their unique characteristics (porous network, low density, light weight, mechanical strength, etc.). Aerogel possess some interesting characteristics like low thermal conductivity (as low as 0.015 W m $^{-1}{\rm K}^{-1}$ ), low density (as low as 1 kg m $^{-3}$ ), high specific surface area (as high as 1000 m $^2{\rm g}^{-1}$ ), and excellent mass transfer properties with coherent porous (porosity more than 95% and pore diameter is in the range from 2 nm to 50 nm) solid structure (Wang et al., 2019a; Wu et al., 2018; Zhang et al., 2017; Zhao et al., 2018; Zheng et al., 2020). Due to these aforementioned characteristics, aerogel grabs the attention of food researchers for the application in food

In 1931, Steven Kistler first developed aerogel (silica gel as precursor material) by increasing the pressure and temperature of the jellies beyond its critical point to replace the liquid of jellies with gas (Yahya et al., 2020; Zheng et al., 2020). The main steps in making aerogel are (i) making hydrogel, (ii) turning hydrogel into alcogel, and (iii) turning alcogel into aerogel. Most hydrogels are made using either the sol-gel method (Chen & Zhang, 2019) or the emulsion gelation method (Kleemann et al., 2018; Selmer et al., 2019).

However, some other gelation method like gelatinization retrogradation (Ubeyitogullari et al., 2018), ionotropic gelation (Plazzotta et al., 2019), heat set (Kleemann et al., 2020; Plazzotta et al., 2020), cold set gel (Ahmadi et al., 2016) and enzymatic cross-linking (Kleemann et al., 2020) can be followed to form hydrogel. Substitution principle [water (present in hydrogel) with alcohol (methanol, ethanol, etc.)] is used to form alcogel from hydrogel. Alcogel are dried (super critical CO<sub>2</sub>, freeze drying, vacuum drying, hot air oven drying, etc.) to form aerogel. It is noteworthy mentioning that freeze drying can convert hydrogel into aerogel directly without converting it into alcogel.

Silica based aerogel are available since last nine decades (approximately). Poly vinyl alcohol (PVA), polysaccharides, proteins, seed mucilage, etc. based aerogel are quite common now a days. Mainly, the trend is going towards bio-based aerogel as these are biodegradable, nontoxic and less hazardous. Protein Kleemann et al. (2018; Plazzotta et al. (2020); Selmer et al. (2019), starch (Ubeyitogullari; Ciftci, 2016; Zhao et al., 2018; Zhu, 2019), and mucilage (Comin et al., 2015; Falahati & Ghoreishi, 2019; Ubeyitogullari & Ciftci, 2020) based aerogel are some of the examples of food grade aerogel.

Aerogel are applied mainly for oil water separation (Li et al., 2017; Meng et al., 2017), adsorption (Chen et al., 2017; White et al., 2014), chromatography (White et al., 2014), thermal insulation (Shang et al., 2017; Yang et al., 2017), ion exchange materials (Keshipour & Khezerloo, 2017; White et al., 2014), air filtration (Zeng et al., 2019), food and drug delivery systems (Bhandari et al., 2017; de Oliveira et al., 2019a), food packaging (de Oliveira et al., 2020; de Oliveira et al.,

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<sup>\*</sup> Corresponding author.

E-mail address: poonam@tezu.ernet.in (P. Mishra).

### **REVIEW**



### Aerogel: Functional Emerging Material for Potential Application in Food: a Review

Subhamoy Dhua1 · Arun Kumar Gupta1 · Poonam Mishra10

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### Abstract

**Objective** The aim of this review is to summarize the production process, types, characteristics, and application of different food-grade aerogels in sensing of food adulterants, and delivery vehicle of bioactive compounds.

Materials and Methods As an emerging material, food-grade aerogels are being used to develop functional food materials (delivery vehicle for bioactive compounds, nutraceuticals, etc. and sensing material for the detection of pesticides, spoilage, etc.). Impregnation of functional compounds in food-grade aerogels have potential in Food Industries.

Results and Discussion Food-grade aerogels exhibit tremendous potential in food packaging-based applications as a moisture absorber, bioactive compound releaser, carrying material, preserver, etc. It also has the potential to serve as target-based delivery vehicle, improves bioavailability of loaded materials, protects them from adverse environment and essential oil incorporated oleogel, etc. Moreover, food-grade aerogels are biodegradable in nature. It is noteworthy mentioning that how the precursor material and production process influences the characteristics of food-grade aerogels also summarized.

**Conclusion** Food-grade aerogels (polysaccharide, protein, and mucilage based) are fulfilling the need for food application due to their exceptional properties (high porosity, high specific surface area, and very low density).

Keywords Aerogels · Preparation · Characteristics · Food-grade aerogels · Applications

### Introduction

In present days, consumers are paying attention to their diet, and they become more health conscious ever before. It is worth mentioning that technology related to food processing has improved over time to fit with the trend accordingly with the consumer's demand. Food scientists and researchers are working irrepressibly to deliver more hygienic,

### Highlights

- Very few studies are present on food-grade aerogels and their applications in food industries.
- Aerogels possess porous structure with good mechanical strength.
- Aerogels are capable of loading, carrying, and releasing of functional compounds.
- Food-grade aerogels are used in food packaging and bioactive compound delivery systems.
- Extensive research on applicability of aerogels in food systems is needed.
- □ Poonam Mishra mpoonam1@rediffmail.com; poonam@tezu.ernet.in
- Department of Food Engineering and Technology, Tezpur University, Assam 784028, India

fresh, safe, nutritious, healthy, and tasty food stuffs to the diverse range of consumers. They are working continuously to improve production process by introducing novel technologies (includes encapsulation and slow release of bioactive compounds), packaging by means of active and intelligent packaging (includes retention of product quality, increased shelf life, preserving and utilizing the characteristics of functional compounds), and safe distribution of food stuffs (Mikkonen et al., 2013; de Oliveira et al., 2019b). To conserve and protect the food from extrinsic factors and to provide health benefits to the consumer, food industries are continuously searching for an emerging technology (de Oliveira et al., 2020).

Aerogel is one of the emerging and important materials among the others (hydrogel, nano-emulsion, etc.). In 1931, Steven Kistler has first developed aerogel (silica gel as precursor material) by increasing the pressure and temperature of the jellies beyond its critical point to replace the liquid of jellies with gas. A series of aerogels (silica, stannic oxide, and cellulose) have been synthesized by Steven Kistler since first development (Zheng et al., 2020). Aerogels have been developed approximately nine decades ago; however, the use of aerogel-based compounds has increased greatly









### NATIONAL CONFERENCE

07

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13-15 February 2025

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Participant/ Poster Presentor / Oral Presenter/ Invitee

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AFSTI at Tezpur University, Tezpur, Assam, India.

Title effect of officeol..... its application in boad.

Co-author(s): U. Popuam Mishea

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> Ramezh Dr. M. Ramesh Naidu Chief Convener

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Department of Soils, Water & Agricultural Engineering, Sultan Qaboos University, Oman in Department of Food Engineering & Technology, Tezpur University, Assam, India, and Association with AFST(I) Tezpur Chapter.

 ${\it litle}$  : effect of humectant (glycerol) on morphology, functional properties of corn starch based aerogel

Authors: subhamoy dhua, poonam mishra

Mach

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Dr. Said Al-Ismaily Chairperson Sultan Qaboos University

> Dr. Pankaj B. Pathare Organizing Secretary Sultan Qaboos University







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Koodwooj K.

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Innovative Food System Transformations for Sustainable Development in Agro-Food

Corn Starch Based Aerogel in International Conference, organized by Of Humectant (Glycerol) On Physical And Mechanical Properties Of Department of Food Technology, VFSTR (Deemed to be University) This is to certify that Mr. / Ms. / Dr. / Prof. Subhamoy Dhua of Tezpur University secured 1st position in poster presentation on topic Effect

Rameh Dr. M. Ramesh Naidu Chief Convener

Dr. Dinkar B. Kamble Organizing Secretary Haubber

Dr. Irshaan Syed Co-ordinator, Food Tech. Sd. Irshaan