7. Summary and conclusion

Developing an emerging material which serves the purpose of conserving and safeguarding the food from external factors is very essential. **Chapter 1** elaborates the introduction to the history, quality characteristics, types, development process, and application of aerogel along with discussions on carbon dots. Techniques and parameters to consider during aerogel development was also discussed briefly. Additionally, the chapter contains brief discussion on CDs and its superior qualities and applications especially in the field of sensing of food contaminants. This chapter also consists of brief discussion on carbon dots incorporated aerogel and their application in food sensing. This chapter also presents the research gap and justification to the necessity of this study.

Chapter 2 expatiates the literature review on polysaccharides, protein, seed mucilage and CDs loaded aerogel. This chapter includes classification of aerogel, precursor required for aerogel development, method of aerogel formation, and their functional properties. This chapter further highlighted the development of functional aerogel from bio-based precursor and their application in different area of food. Development method and application of corn starch based aerogel was also reported in this chapter. This chapter also elaborated about synthesis of CDs, functional properties and application of CDs. Alongside, discussion on methods of CDs loading in aerogel matrix, CDs based aerogel development, characterization of CDs based aerogel and their application in various area of sensing like, recognition of NO_x and detection of aldehyde species (glutaraldehyde) in water, detection of UO_2^{2+}, Sm^{3+}, and Eu^{3+} in ground water was also reported in this chapter. Alongside, conventional and advanced methods of formalin detection have been discussed in detail.

Chapter 3 covers the detailed development process (preparation of hydrogel, alcogel through ethanol substitution, and $SCCO_2$ drying of alcogel) of aerogel, characterization in terms of physico-functional (density, porosity, water absorption capacity, hygroscopicity, etc.) mechanical (compressive strength and recompressibility), thermal, and morphological properties and application in the storage of fresh spinach leaves. The impact of glycerol on the quality characteristics have discussed in this chapter. Glycerol-infused aerogel had a more connected, denser structure (0.38–0.45 g/cm^3)due to development of a thick and more connected network (due to the formation of hydrogen bonds between polymer chains upon efficient action of glycerol) within the internal matrix of aerogel, enhanced hygroscopic behaviormay be attributed to the inherent affinity of both starch and glycerol toward the water, and was reusable up to eight times in terms of its capacity to absorb water after being drawn from the soaked sample. The addition of glycerol might enhance the site where water molecules can bond, which could scavenge a sizable amount of moisture. The inclusion of glycerol reduced the aerogel's porosity (75.89–69.91 %) and water absorption rate ($WAC_{30\ min}$; 118.53–84.64 %) which may be due to their dense structure, that prevents quick water absorption through the capillary system. The enhanced compressive strength (26.01–295.06 N) may be due to a more compact structure developed after glycerol addition, resulting in a higher strain. The increased value of crystallinity due to the addition of glycerol which may increase the alignment of polymer crystals and form a more regular and crystalline area with sharp peaks. These properties will help to find a broad area of its potential application like sensing, moisture absorber, repeated use in water, smart packaging, etc.

Chapter 4 detailed the development of aerogel using different drying techniques (freeze drying and microwave drying). Microwave drying exhibited comparable results in terms of physico-functional, mechanical, morphological, etc. which supports this technique as a new way of aerogel development from the bio-polymer based precursor materials. Glycerol addition showed a negative impact in the freeze dried aerogel while it showed positive impact on freeze dried aerogel in properties like morphological, mechanical, thermal, and reusability. However, porosity was reduced considerably in microwave dried aerogel having high glycerol content (10 %) which results in low water absorption capacity as the vacant spaces get reduced. The crystallinity of aerogel was increased as a result of glycerol addition. Microwave dried aerogel exhibits nano range pores which can find its application in bioactive and drug delivery systems.

Chapter 5 deals with the development of CDs based aerogel. The aerogel was first developed through microwave drying (hydrogel→ alcogel→ aerogel) approach. The CDs was synthesised through hydrothermal method using citric acid monohydrate and ammonium hydroxide as precursor solution. The solution was put in a Teflon lined autoclave and was kept at 200 °C for 5 h for CDs synthesis. Characterization of synthesised CDs was done in terms of yield (0.60 %), quantum yield (≈ 46 %), particle size (2.21 nm), FTIR, NMR, UV-absorbance, fluorescence intensity, etc. The aerogel was dipped in the CDs solution for overnight to get loaded inside the aerogel matrix. Then the wet aerogel was dried through freeze drying (-50 °C, 3 h) to get CDs loaded aerogel. Alongside, the characterization of CDs loaded aerogel (loading value, FTIR, G/B value, thermal properties, and XRD) was also done. The developed

CDs based aerogel have retained stable fluorescence characteristics as exhibited by CDs. The CDs based aerogel showed uniformity in CDs dispersion inside the aerogel matrix. The FTIR spectra confirmed that the CDs based aerogel have retained both the native qualities of corn starch and CDs. The aerogel was used as pH sensor. The aerogel was calibrated with different pH tuned CDs solutions, then the G/B value was obtained. A calibration curve of G/B values against different pH was made to get an unknown from its G/B value. The CDs based aerogel exhibited good results with a deviation of only 9% while determining pH through spiking approach.

Chapter 6 elaborates on the application of CDs loaded functional aerogel as formalin detector and its validation with the existing acetylacetone method. The detection was based on the silver mirror method. TR was used in combination with CDs to execute the sensing mechanism. The calibration of CDFA involved addition of standard FA solution on CDFA. The calibration curve was used to detect the presence of formalin in fish. The CDFA exhibited LOD and LOQ of 5.55 mg/L and 18.50 mg/L with minimal bias (- 0.39 % Bias) and variation (4.71 % RSD). It achieved a recovery percentage of 100.39 – 102.02 % for standard FA solutions. The performance of the CDFA was validated with the % deviation values with reference to acetyl acetone method. It was observed that % deviation ranges from – 6.24 to + 10.28 %, when the experiment conducted with the extract obtained from fish fillets. The % deviation was observed to be varied from – 11.72 to + 7.98 % for the extract obtained from whole fish. These findings supported the compatibility of CDFA in real sample analysis.

All the objectives together strengthen the development of CDs based functional aerogel and its compatibility in detecting FA in fish besides representing itself as a pH sensor.

Contribution to the knowledge

1. Lightweight, porous, mechanically stable corn starch-based aerogel is developed.
2. Addition of glycerol increases the number of interconnections inside the aerogel matrix which improved the mechanical strength of aerogel.
3. Microwave drying can be adopted as an alternative and sustainable drying technique in the area of development of corn starch based aerogel.
4. Glycerol added aerogel is reusable up to 8 times except for freeze dried aerogel.
5. The developed aerogel is loaded with CDs to fabricate a multifunctional aerogel.
6. CDs based aerogel is successfully utilized for determination of pH.
7. Fabricated CDs based functional aerogel (CDFA) for sensing of formalin.

8. The developed method is rapid, portable, and can sense the FA with limit of detection (LOD) of 5.55 mg/L.
9. The developed CDFA is specifically suitable for determination of formalin in food sample.
10. The developed method is successfully validated with the conventional method (acetylacetone method).
11. This method of FA detection is easy as compare to the conventional detection methods.
12. The CDFA can be easily used by the public to detect FA in fish and to restrict illegal adulteration of fish with FA.

Limitations and Future scope

Beyond the contribution of aerogel, it possesses some limitation which restricts its wide application are as follows:

1. Development of aerogel itself is a very time consuming process and proper handling of material at every stage of aerogel preparation is highly needed.
2. CDFA can only be used for single use, afterthat it was difficult to bring it to its initial stage without significant change in the quality of the CDFA.
3. CDFA cannot be stored in a normal light and environmental conditions for a long period of time as it start oxidizing rather it can be stored in very less humid and dark conditions.

There are further scope of research beyond this work which can be done to develop a functional aerogel with more practical significance:

1. The aerogel can be used to detect formalin in vegetables, other fresh water fishes and sea fishes etc.
2. The aerogel can be modified to detect other contaminants.
3. CDs can be tuned to enhance its function specific characteristics so that it can be loaded to perform target specific actions.
4. Bio-waste based sources of CDs can be used to promote waste management.
5. The CDFA can be modified in such a way that it can perform multiple operation.