Biological Forum – An International Journal **15(10): 695-698(2023)**

ISSN No. (Print): 0975-1130
ISSN No. (Online): 2249-3239

Development of Chitosan based Electrospun Nanofibers for Active and Intelligent Packaging

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(Received: 06 August 2023; Revised: 02 September 2023; Accepted: 26 September 2023; Published: 15 October 2023)
(Published by Research Trend)

ABSTRACT: The active and intelligent packaging market is rapidly growing, driven by the increasing demand for fresher, higher quality food at reasonable costs. Nanotechnology, with its unique properties and potential applications, has emerged as a promising field for improving food packaging. Electrospinning, a simple technique for producing ultrafine polymer fibers, is particularly relevant in this context. Chitosan, a biocompatible, biodegradable polymer, has shown remarkable potential in the development of electrospun nanofibers for intelligent packaging applications. This study focuses on the development of chitosan-based electrospun nanofibers and their suitability for smart packaging. Optimal conditions for achieving smaller diameter nanofibers were identified as 1.0 mL h^{-1} flow rate and 25 kV of voltage.

Keywords: Electrospinning, Flow rate, Voltage, Viscosity and Surface tension, Chitosan, Nanotechnology.

INTRODUCTION

The global active and intelligent packaging market is expected to grow at a compound annual growth rate (CAGR) of more than 4% during 2018–2024. The active and intelligent packaging market was valued at USD 17.5 billion in 2019 and projected to grow to USD 25.16 billion by 2025, at a CAGR of 6.78% during the forecast period from 2020 to 2025 (www.mordorintelligence.com). The global food market has a higher than ever demand to deliver fresher, quality food at a reasonable cost (Nicoletti and Serrone 2017). Hence, current food processing practices and technologies must be assessed and improved.

Nanotechnology is one of the emerging research areas with tremendous scientific and economic potential. It focuses on the characterization, fabrication and manipulation of structures smaller than 100 nm in at least one of the dimensions. Structures on this scale have unique and novel functional properties. Electrospinning (ES) is one of the simplest techniques that applied in the preparation of ultrafine micro and nanometer diameter polymer fibers using high electric fields (Wang *et al*., 2009). These fibers can be matrixed in the form of a non-woven mat or as nano membrane (Agarwal *et al*., 2012).

Electrospinning can be used to produce various functional fibers with many advantages like low cost, easy tuning of morphology and capacity to produce continuous long fibers (Reneker and Chun 1996). It is preferred over solvent casting, centrifugal spinning, melt blowing and phase separation which are in fact the mechanisms that are used in production of traditional films and nanofibers, because the nanofibers produced by electrospinning possess an extremely high specific surface area, high porosity, small pore size and high absorbance capacity which render them highly suited to be used in chromic indicator (Greiner and Wendorff 2007). The high surface to volume ratio of these fibers has attracted their applications in sensors. The pH sensitive electrospun nanomembranes can find wide applications in the areas of smart packaging of food and beverages, textile, tissue engineering, water filtration, studying microenvironment in various biological process, microbiological studies, *etc*., where pH change is involved (Tassanawat *et al*., 2007).

Chitosan (C) $C_6H_{11}NO_4$ is a naturally abundant and renewable polymer and has a number of properties that make it ideal for electrospinning nanofiber, including: biocompatibility, biodegradability, good film forming ability and high surface area (Qasim *et al*., 2018). Electrospun chitosan nanofibers have a very high surface area-to-volume ratio. This makes them very good at absorbing fluids and other molecules, making them ideal for applications such as intelligent packaging and smart packaging. Chitosan can form strong and flexible films, which are ideal for electrospinning nanofiber. By manufacturing chitosan into nanofibers using the electrospinning method, its potential is amplified due to the enhancement of the active surface and the low preparation cost (Anisiei *et al*., 2021). Once disposed of, the chitosan does not take long time to degrade and is safe for environment than petroleum based plastics (Jiffy *et al*., 2013).

MATERIALS AND METHOD

The selected materials exhibit distinct characteristics that align with the objective of the selected study, ensuring the development of biodegradable nanofiber mat. For the present study, the biopolymer chitosan was procured from M/s. Enzyme India Private Limited, Chennai. Chitosan solution was prepared using concentrated aqueous acetic acid solution. Chitosan (CAS 51570-20-80), a base material was added at concentration of 7% w/v per 100 mL of 90% v/v aqueous acetic acid solution. The solution was heated at 70°C and stirred at 1000 rpm using digital magnetic stirrer for 2 h to obtain uniform dissolution of chitosan in solution (Geng *et al*., 2005). The chitosan solution was electrospun at different flow rate (0.5, 1.0 and 1.5 mL h^{-1}) and voltage levels (20, 25 and 30 kV). The process for preparation of chitosan based electrospun nanofiber mat depicted in Fig. 1.

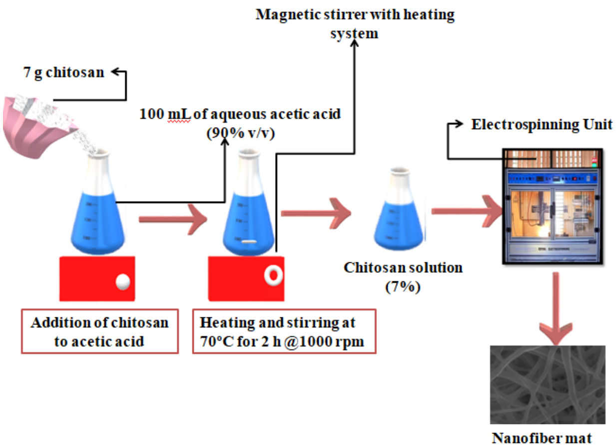


Fig. 1. Process for preparation of chitosan based electrospun nanofiber mat.

A. Electrical conductivity

Electrical conductivity (EC) of chitosan solutions was determined by following ASTM (D1125-23, 2005) using electrical conductivity meter (Water Analyser 371, Systronics, Ahmedabad, India) at 28ºC±2. The experiment was replicated thrice and average electrical conductivity was measured and expressed in µS.cm^{-1} (Duan *et al*., 2023).

B. Viscosity

Viscosity of chitosan solutions was determined by using modular compact rheometer (Anton Paar GmbH. MCR102, Graz, Austria). The experiment was replicated thrice and average viscosity was measured and expressed in Pa. s (Maftoonazada and Ramaswamy 2019).

C. Surface tension

Surface tension of the indicator solution was determined by drop count technique by using stalagmometer (Isko, Boro 3.3. Ambala, Haryana, India).

D. Colour value

Colourimeter (Colour Flex EZ, Hunter Associates Laboratory, United States) was used for the measurement of colour values of indicator solution. The colour values were measured by using CIELAB scale at 10° observer at D_{65} illuminant. It works on the principle of focusing the light and measuring energy reflected from the sample across the entire visible spectrum. It provides reading in terms of L^*, a^* and b^* values. Where, luminance (L^*) forms the vertical axis, which indicates whiteness (+) to darkness (-). In the same way a^* indicates redness (+) to greenness (-) and b^* indicates yellowness (+) to blueness (-).

E. Electrospinning for the development of chitosan nanofiber mat using chitosan solution

Electrospun nanofiber mat was developed using a collector and plate electrospinning machine (Royal Enterprises, MD405, Chennai, India).

F. Diameter and morphological characterization

The diameter and morphological characteristics of developed electrospun nanofibers was studied by using scanning electron microscope (Carl Zeiss Microscopy, EVO 10, Cambridge, UK). At least 100 nanofibers were randomly selected from each of the SEM images and the diameter size distribution was determined using the Image J software (Wen *et al*., 2016).

RESULTS AND DISCUSSION

A. Electrical Conductivity

Electrical conductivity of chitosan solution was 125.333±1.453 µs. cm^{-1}. Electrical conductivity of biopolymer solution is an important variable; minimum voltage and maximum electrical conductivity would form the good quality nanofibers. This phenomenon allows electric stretching forces to surpass the surface tension of the polymer solution, particularly in low electric field conditions. Consequently, this enables the production of uniform nanofibers.

B. Viscosity

The viscosity of chitosan biopolymer solution was 1149.533±0.481 mPa. S. The viscosity of the solution is a major factor that determines the fiber diameter in electrospinning process. The viscosity of the electrospun biopolymer solution must be within an acceptable range in order to achieve electrospinning

(Luzio *et al*., 2014). Similar results were reported by Amariei *et al*. (2017) for electrospinning solution with a viscosity of 100 to 2000 mPa.s to be suitable for electrospinning.

C. Surface tension

The surface tension of a liquid is a measure of its cohesiveness, or the tendency of its molecules to stick together. A high surface tension indicates that the liquid molecules are strongly attracted to each other, while a low surface tension indicates that the liquid molecules are more easily separated (Jiang *et al*., 2004). The surface tension of chitosan solution was 56.62756.627±0.067 dyne cm$^{-1.}$ The surface tension values recorded are within the range of 35 to 58 dyne.cm^{-1} for electrospinning for polymeric solution for generating uniform nanofibers (Amariei *et al*., 2017).

D. Colour values

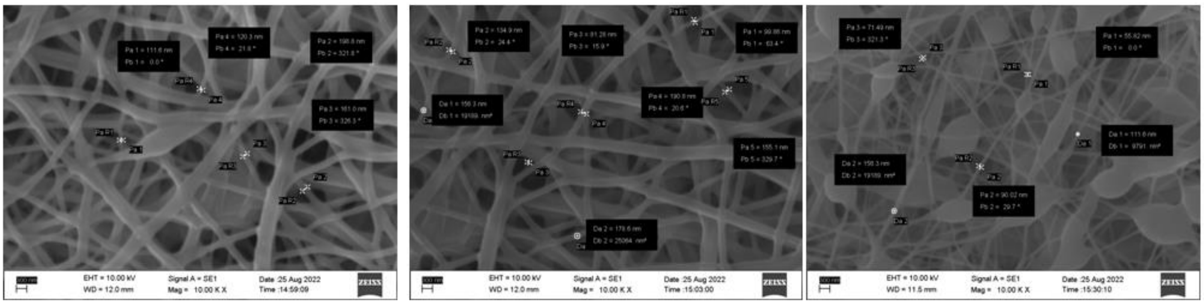
The *L** value is a measure of lightness, with a higher value indicating a lighter colour. Chitosan biopolymers solutions showed higher brightness value of 86.233±0.026. The *a** and *b** value of chitosan solution was -2.300±0.058 and 12.420 ±0.420, respectively. The colour value indicates the chitosan solution exhibits slightly yellow colour with glassy brightness. Similar results were obtained by polyvinyl alcohol/chitosan, where the *L** value, which was 62.71.

Table 1: Properties of chitosan solution for electrospinning.

Sr. No.	Properties	Results
1.	Electrical Conductivity (µs. cm^{-1})	125.333±1.453
2.	Viscosity (mPa. s)	1149.533±0.481
3.	Surface tension (dyne. cm^{-1})	56.627±0.067
4.	Colour value *L** value *a** value *b** value	 86.233±0.026 -2.300±0.058 12.420±0.420

E. Diameter and morphological characterization

One hundred nanofibers from each mat were considered as a mean diameter of the nanofiber mat. Diameter of nanofiber is one of the most important parameters of electrospinning and is greatly affected by solution viscosity, surface tension, voltage and flow rate of the polymer (Ojha, 2017). The lower diameter nanofibers have more surface area, better mechanical properties and more uniform morphology. It was observed that the diameter of nanofiber was significantly affected by the flow rate as well as voltage. It was observed that the lowest diameter nanofibers were obtained at 1.0 mL. h^{-1} and 25 kV of voltage i.e., 190±0.521nm and highest nanofiber diameter was obtained at 1.5 mL.h^{-1} floe rate and 30 kV with size of 210.065±0.121 nm. But at higher flow rate and voltages there was a formation of solution droplets. Similar results were obtained for poly (vinylidene fluoride) at lower flow rate (0.02 mL. min^{-1}) induces the formation of beads defects and produce fibers with cylindrical morphology (Zulfikar *et al*., 2018).



0.5 mL. h^{-1} and 20 kV   1.0 mL. h^{-1} and 25 kV   1.5 mL. h^{-1} and 30 kV

Table 2: Effect of processing parameters on morphology of nanofiber.

Sr. No.	Flow rate	Voltage	Nanofiber diameter
1.	0.5	20	201.656 ±0.144 nm
2.	1.0	25	190.000±0.521nm
3.	1.5	30	210.065±0.121

CONCLUSIONS

The development of chitosan based electrospun nanofibers for intelligent packaging holds significant promise for enhancing the active and intelligent packaging market. Electrospinning of chitosan offers a sustainable and biodegradable solution with properties like biocompatibility, biodegradability, and a high surface area-to-volume ratio, making it an ideal candidate for applications in smart packaging. The experimental results have provided insights into the key parameters for successful electrospinning of chitosan nanofibers. The electrical conductivity, viscosity, and surface tension of the chitosan solution play crucial roles in determining the quality of nanofibers. Furthermore, the diameter and morphological characterization of the electrospun nanofibers showed that the choice of flow rate and voltage significantly influences the nanofiber size and morphology. The most favorable conditions for obtaining smaller diameter nanofibers were at a flow rate of 1.0 mL. h^{-1} and a voltage of 25 kV. In the context of intelligent packaging, these electrospun chitosan nanofibers exhibit great potential for various applications, including pH-sensitive indicators, food and beverage packaging, and other areas where pH changes are involved. The development of eco-friendly, biodegradable nanofiber materials aligns with the growing demand for sustainable packaging solutions in the food industry. The use of chitosan-based electrospun nanofibers opens up new possibilities for innovative and environmentally friendly packaging solutions, contributing to the evolution of active and intelligent packaging technologies to meet the demands of the global food market.

FUTURE SCOPE

Incorporating electrospun nanofibers with active ingredients such as antimicrobial agents, antioxidants, or oxygen scavengers can help extend the shelf life of packaged products. Electrospun nanofiber-based sensors and indicators can be integrated into packaging materials to monitor freshness, temperature, humidity, or gas concentration inside the package. This can help consumers and producers make informed decisions about the safety and quality of the packaged products.

Acknowledgement. The authors are thankful to Center for Nanotechnology and Department of Processing and Food Engineering providing conducting and analyzing the experimental study

Conflict of Interest. None.

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How to cite this article: Shourathunnisa Begum, Sharanagouda Hiregoudar, Udaykumar Nidoni, Ramappa K.T., Kurubar A.R. and Saroja N. Rao (2023). Development of Chitosan based Electrospun Nanofibers for Active and Intelligent Packaging. *Biological Forum – An International Journal, 15*(10): 695-698.