STUDY OF OPTICAL, STRUCTURAL AND DIELECTRIC PROPERTIES OF SWIFT HEAVY ION IRRADIATED BIODEGRADABLE POLYMERIC MATERIALS

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Preface

At present, the demand for production and fabrication of polymeric materials are gain worldwide due to its necessity in our daily life. However, the use of synthetic polymers promote the challenges of waste disposal and environmental risk. Moreover, the process of plastic recycling reveals a complex, challenging and a negative eco-balance because of waste of water as well as energy consumption. As a result, plastic industries to the citizens need the alternative of the conventional polymer. So, the research communities from the worldwide concern significant efforts and objectives concerning *Biodegradable polymers*- as an alternative to conventional polymer owing to its eco-friendly nature. According to ASTM standard D-5488-94d and European norm EN 13432, "biodegradable" means "capable of undergoing decomposition into carbon dioxide, methane, water, inorganic compounds, and biomass" [1]. Biodegradable polymers can be mainly classified as natural polymers (starch, chitin, chitosan, protein) and biodegradable polyesters (polyhydroxyalkanoates, poly(lactic acid)) [2].

Chitosan is a natural biodegradable and biocompatible, deacetylated polysaccharide derivative from second most abundant polymer chitin, extracted from the shells of crabs, shrimp and prawns. The chitosan is the only pseudonatural cationic nature polymer with reactive functional amine ($–NH_2$) and hydroxyl (–OH) groups allow versatile modifications for desired applications in many fields [3]. Chitosan used as a host matrix, due to its excellent film-forming ability and ease of processability. Moreover, the physicochemical properties of chitosan being tailored extensively in various fields by incorporating organic (another polymer, plasticizer, agro-additive, etc.) and addition of inorganic (salt, nanoparticles, ceramic, etc.) fillers as per necessities. However, the chitosan matrix grafted the filler up to a certain extent.

Firstly, blending of chitosan with numerous polymers [4] being synthesized and used in different fields. The blending of chitosan with Poly(ethylene oxide), PEO, is being widely investigated [5–7]. PEO is a water-soluble, non-toxic, biodegradable linear polyether. PEO forms lamellae which are arranged in spherulites and the degree of the crystallinity is about 70–85% [6]. However, low flexibility and comparatively weak mechanical properties of crystalline PEO and poor conductivity of chitosan constrain its various applications. By blending these two polymers enhance the overall performance of blend matrix [7] and used in biomedical, pharmaceutical, food, textiles [6] sectors as well as polymer host in batteries, super capacitor and electrolytes applications [8,9].

Moreover, the chitosan biopolymer is used as a host matrix for solid polymer electrolytes (SPEs). Due to functional groups with oxygen and nitrogen atoms possess a lone pair of electrons, which help chitosan to form complexes with the salt. The SPEs system have certain advantages over liquid electrolytes no leakage and corrosion, flexibility, low weight, transparent, ease of processing and operative at higher temperature [10]. Hence, they are being studied for technological development in the field of batteries, super capacitors, sensing devices, fuel cells, electrochemical cell, etc. [11]. The SPEs having silver ion charge carriers used for longer period exhibited poor stability in terms of Ag^{+} to Ag^{0} transformation. Hence, it should be desire to use the silver-based salt in SPEs with high lattice energy. Silver nitrate ($AgNO_3$) salt having lattice energy 773 kJ mol^{-1} operated at moderate temperature hindered Ag^{+} to Ag^{0} transformation [12]. In comparison with other metal-ions conducting salts, the silver nitrate has an advantage of good complexation with polymeric matrix, eco-friendly, low-toxic and non-inflammable.

The recent innovations and developments in the fields of flexible and green electronics led to fabricate the multifunctional flexible materials with desire dielectric properties [13]. Also, this kind of materials should be to low-weighted, bendable, compact, eco-friendly, low-cost and ease of processing. All such requirement can be fulfilled by using biodegradable polymer. However, the dielectric permittivity of such polymer is moderately low for electronics utilizations. This constrains could be overcome by incorporating various types of filler viz. ferroelectric, ceramic or metallic nanoparticles (NPs) [14–16] called biodegradable polymer nanocomposites (BPNs). BNP is two-phase materials in which one of the phases has at least one dimension in the nanometer range (1–100 nm) [17]. BPNs are widely utilized in the field of electronics, environment, optics, medicine, wearable devices, capacitors, batteries, sensor, electromagnetic shielding etc. Chitosan has an excellent chelating mechanism could interact with many metallic NPs such as gold, silver, platinum, and palladium [18]. Among various metallic NPs, silver nanoparticles (AgNPs) are vital and fascinating nanomaterial with unique physicochemical properties like decent electrical conductivity and surface plasmonic resonance (SPR) behaviour having a broad spectrum of applicability [19,20].

As discussed, chitosan/PEO (CP) blend has an improved superior responses over individual polymer shortcomings. The miscibility or compatibility of polymers depends on functional group, polymer composition, molecular weight and additives level. Incompatibility between two polymers may exist due to feeble interfacial adhesion, different polarities,

particular group interactions, and the difference in molecular weight [21]. Consequently, to prepare the polymeric blend with optimum responses, the various compatibilizing agent is being used viz. organic (i.e. block copolymers, plasticizer, reactance) and inorganic (i.e. layered silicate, metallic nanoparticles, carbon nanotubes) fillers. By dispersing the AgNPs in CP blend may interact with functional groups of individual polymer yielding the affinity of the polymers for each other. Hence, the structural, optical and dielectric properties of the biodegradable polymer blend upon addition of NPs significantly changed as a function of additive level [22].

To date, chitosan is being characterized and modified via physical and chemical methodologies according to necessity in vast fields worldwide. The modifications of chitosan as a function of additive either organic or inorganic could be implemented up to a certain extent of the additive level. Moreover, several other methodologies used in modifications of chitosan implicated various parameters and hence it is become complicated to explain and correlates various parameters. Hence, it is being necessitated to modified the biodegradable polymeric matrix via well-controlled technique in constraining environmental conditions. An advanced and versatile technique to alter the structural, optical and dielectric responses for required applications is radiation treatment. The radiation classified as non-ionizing radiations or ionizing radiations depending on energy. The radiation has enough energy to perturb the materials through which it passed by displacing or vibrating the atoms of the molecule without removing the electron is referred to as non-ionizing radiation. The radiation having abundant energy able to knock out the orbital electron of the atom and creating ions along its trajectory within the materials is referred to as ionizing radiation. Ionizing radiation, viz. electromagnetic (e.g. UV, X-ray or Gamma-ray) or particle (electron, neutron or charged species) interaction with biodegradable polymeric materials significantly modified the matrix by depositing the energy in materials at macro-level. However, the deposited localized energy density to the matrix by electromagnetic radiation is rather small and hence it may transform the materials up to a certain extent through photooxidative degradation. Swift heavy ions (SHIs) moving at a velocity comparable to the Bohr velocity of the electron [23] and depositing tremendously high localized energy density within a very small volume ($\sim 10^{-17}$ to 10^{-16} cm^3), in a very short time ($\sim 10^{-17}$ to 10^{-15} s) [24]. Moving SHIs of energy in order of MeV through polymeric system forming molten cylindrical zones having ions, electrons, radicals and polymeric chains. As polymer matrix return to its equilibrium solid form yielding macroscopic changes viz. structural, optical, dielectric, surface, thermal properties

etc. [25].The polymeric matrix with desired properties can be engineered by the informed choice of the energy, mass, and fluence of the SHIs and the level of modifications depends upon electronic energy loss (S_e), atomic number and diameter of ion track as well target itself.

Sionkowska et al. [26] studied the surface properties of chitosan films upon UV-irradiation. Ulanski et al. [27] investigated optical changes in chitosan in solid state and in aqueous solution upon Gamma irradiation. Lim et al. [28] examined structural, mechanical and thermal properties of Gamma irradiated chitosan. Ramnani et al. [29] explored gamma ray induced crosslinking of chitosan in the presence of carbon tetrachloride. Garcia et al. [30] reported the correlation between reduced molecular weight with the antioxidant capacity of chitosan upon gamma irradiation. Wanichapichart et al. [31] studied low-energy (15–30 keV) nitrogen ions induced filtering characteristics modification of chitosan membrane. Kim et al. [32,33] reported chemical, physical and molecular weight alteration of chitosan upon proton irradiation (30–40 MeV). Prakrajang et al. [34] described Ar and N ions (15–25 keV) induced changes in surface morphology, contact angle and electric characteristics of chitosan membrane. Gryczka et al. [35] discussed the degradation mechanism of chitosan upon gamma and electron beam irradiation. Wanichapichart et al. [36] studied the influence of membrane permeate fluxes and microbial growth upon Ar and N ions (30–120 keV). Yulianti et al. [37] reported the synthesis of chitosan-based polymer electrolytes through ion implantation approach.

Yang et al. [38] revealed a change in crystallinity and molecular mass upon Gamma irradiation of PEO. Zhang et al. observed amorphization of PEO at a high dose of Gamma-ray. Schilling et al investigated the dynamics of PEO chain using DSC and NMR upon Gamma irradiation. Nedkov et al. [39] reported that the thermal response of PEO is governed by induced fractions upon Gamma irradiation. Doytcheva et al. [40,41] studied UV radiation-induced crosslinking of solid PEO as well as PEO with tetraalkyl ammonium salt. Zainuddin et al. [42] revealed that degradation of PEO dominates up to 15 kGy and for higher dose crosslinking gains upon Gamma irradiation. Jurkin et al. [43] explored Gamma-ray induced modification in the physical properties of PEO/silica films and gels. Deka et al. [44] observed an enhancement in dielectric properties of PEO/montmorillonite matrix upon oxygen ion (90 Mev) irradiation. Kishore et al. [45] perceived modification in the physicochemical properties of electroactive $PEO_{25}NaI$ + 15% SnO_2 system upon silicon ion (120 MeV).

Kowalonek [5] recently discussed photooxidation of CP blend upon UV irradiation. He observed that the alteration in surface and thermal properties owing to irradiation depends on blend composition. Kianfar [46] et al. explored thermal, morphology and water resistance properties of electrospun upon UV treatment of CP blend obtained by electrospinning method.

To date, enormous efforts dedicated towered the modifications of biodegradable polymeric matrixes viz. chitosan, PEO, chitosan/PEO involving organic and inorganic additives in research as well as industrial sectors. Also, they are being modified using UV, Gamma-rays and low energetic ions. As per the literature review, the information is still lacking about the high energetic SHIs irradiation induced effects on biodegradable polymeric materials, in spite of its increasing importance for applications in interdisciplinary fields. The work planned here as a part of the thesis is aimed to study the effects of SHIs irradiation on biodegradable polymeric materials as a function of beam parameters.

Objective

In the present investigation, we have prepared three systems based on chitosan and PEO host matrix via solution casting technique. The self-sanding pristine polymeric films of (i) chitosan and CP blend (ii) Biodegradable SPE matrix based on chitosan with silver nitrate salt at different concentrations and (iii) Biodegradable polymeric nanocomposites based on chitosan and CP blend with silver nanoparticles were prepared in the laboratory. The effect of mass, energy, electronic energy loss (S_e) and fluence of the ion beam on structural, optical and dielectric responses of prepared systems was investigated. We have chosen lighter ion C^{+5} (60 MeV) and heavier Ni^{+7} (100 MeV) ion for the irradiation. This will provide better insight and interpretation about the interaction of SHIs with biodegradable systems as a function of beam parameters. The effects of MeV ions on optical, structural and dielectric properties of the biodegradable pristine polymer, biodegradable electrolyte and biodegradable nanocomposites matrixes have been investigated and discussed in the following chapters;

Chapter 1: Introduction

The brief overview of biodegradable polymers and importance of chitosan-based biodegradable polymeric materials. Modification of chitosan by different methods viz. chemical, physical and irradiation have been included. Also, the type of radiation and interaction of SHIs with the matter have been discussed. In-depth literature review of

modification of proposed chitosan-based polymeric materials by ionizing radiation and their development is presented along with the objective of the present investigation.

Chapter 2: Experimental Method

This chapter described the properties, structure and fundamental applications of the biodegradable polymeric materials used in the present investigation. Preparation of self-sustained polymeric matrixes, as well as the working principle and operations of Pelletron accelerator used for the perpose of controlled MeV ions irradiation in constraint environment at Inter University Accelerator Centre (IUAC), New Delhi, India, have been disscussed. Also, the electronic energy loss and nuclear energy loss of MeV ions with proposed matrixes have been obtained by simulating SRIM code. To investigate the tailored optical, structural and dielectric responses of biodegradable matrixes under investigation have been exploited by using UV-Visible, XRD, FTIR, Impedance analyser and AFM/SEM techniques.

Chapter 3: Modification of Biodegradable Polymeric Matrices by Irradiation with MeV Ions

This chapter belongs to investigate the effect of MeV ions on pristine biodegradable polymeric films viz. chitosan and CP blend as a function of beam parameters. These films were irradiated with lighter C^{+5} (60 MeV) and heavier Ni^{+7} (100 MeV) ions at different fluencies. Modification in optical and structural responses have been discussed and well collaborated with frequency dependent dielectric spectroscopy at room temperature.

Chapter 4: Modification of Chitosan-based Biodegradable Solid Polymer Electrolyte by Irradiation with MeV Ions

This chapter focused on the characterization of silver nitrate doped chitosan for electrolyte applications. Prepared SPE was studied as a function of additive and beam parameters. These films were irradiated with lighter C^{+5} (60 MeV) and heavier Ni^{+7} (100 MeV) ions at different fluencies. Optical alteration upon irradiation has been studied by employing Tauc and Fink approaches. Complexation of chitosan with salt and structural rearrangement upon radiation treatment have been studied by using XRD and FTIR spectroscopy. Modification in the relaxation process and conductivity of electrolyte of post-irradiated matrixes have been investigated by dielectric spectroscopy.

Chapter 5: Modification of Chitosan and CP Blend-based Biodegradable Polymer Nanocomposites by Irradiation with MeV Ions

This chapter describes the characterization of two BPNs prepared by dispersion of AgNPs conductive fillers at a different additive level in chitosan and CP blend matrixes.

These films were irradiated with lighter C^{+5} (60 MeV) and heavier Ni^{+7} (100 MeV) ions at different fluencies. Comparative study of these systems for pristine and post-irradiated self-standing matrixes have been discussed. MeV ions induced alteration in various properties have been studied by employing different techniques.

Chapter 6: Conclusions and Future Scope

This chapter summarizes outcomes of C^{+5} (60 MeV) and Ni^{+7} (100 MeV) ions irradiated chitosan-based biodegradable matrixes along with salient conclusions of the investigation. Future perspectives for further research are also expressed.

List of Research Paper Published in Peer-Reviewed Internation Journals

1. SHI induced modification in structural, optical, dielectric and thermal properties of poly ethylene oxide films
   Gnansagar B. Patel, S. Bhavsar, N.L. Singh, F. Singh, P.K. Kulriya,
   Nucl. Instruments Methods Phys. Res. Sect. B Beam Interact. with Mater. Atoms. 379 (2016) 156–161.
   doi:10.1016/j.nimb.2016.04.018.

2. Modification of chitosan-based biodegradable polymer by irradiation with MeV ions for electrolyte applications
   Gnansagar B. Patel, N.L. Singh, F. Singh
   Mater. Sci. Eng. B. 225 (2017) 150–159.
   doi:10.1016/j.nimb.2019.03.052.

3. Effects of MeV ions on physicochemical and dielectric properties of chitosan/PEO polymeric blend
   Gnansagar B. Patel, N.L. Singh, F. Singh, P.K. Kulriya
   Nucl. Instruments Methods Phys. Res. Sect. B Beam Interact. with Mater. Atoms. 447 (2019) 68–78.
   doi:10.1016/j.nimb.2019.03.052.

List of Other Research Paper Published in Internation Journals

1. Investigation of optical properties of aluminium oxide doped polystyrene polymer nanocomposite films
   S. Bhavsar, **Gnansagar B. Patel**, N.L. Singh
   Phys. B Condens. Matter. 533 (2018) 12–16.
   doi:10.1016/j.physb.2017.12.055.

2. Effect of γ-irradiation on optical properties of Eu_2O_3-doped polystyrene polymer films
   S. Bhavsar, **Gnansagar B. Patel**, N.L. Singh
   Luminescence. 33 (2018) 1243–1248.
   doi:10.1002/bio.3541.

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