Research on Polyethylene Glycol, Crosslinked Polyethylene Glycol & Polyethylene Glycol Chitosan Conjugate Coating for Biomedical Application

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Abstract—Polyethylene glycol used as coating materials because it resist to protein adsorption and bacterial adhesion. Polyethylene glycol and crosslinked polyethylene glycol & polyethylene glycol chitosan conjugate used for coating glass implants. Fourier transform infrared spectroscopy (FTIR) analysis showed proper crosslinking in crosslinked polyethylene glycol but in polyethylene glycol & chitosan conjugate no reaction takes place. Scanned Electron Microscope (SEM) used for study of surface morphology. Polyethylene glycol & chitosan conjugate have smoother surface than pure polyethylene glycol, polyethylene glycol chitosan conjugate. Differential scanning calorimetry (DSC) analysis used for glass transition temperature study. Crosslinked polyethylene glycol has good mechanical properties. Antimicrobial properties of polyethylene glycol chitosan conjugate are good in comparison to pure polyethylene glycol, crosslinked polyethylene glycol.

Keywords: Chitosan, Polyethylene glycol (PEG), Antimicrobial properties, Mechanical testing, Thermal properties.

I. INTRODUCTION

Biomaterials are widely used in many implants and medical associated devices; to be implanted inside human body it should have excellent bulk as well as surface properties. The use of these medical devices has lead to new field of revolution in field of medicine. Increased use of these devices has not only increased the life expectancy of patient but also resulted in better quality of life. But one of the major barriers in long term usage of these devices has micro infection. The only solution to this problem is removal of implants which increase patient mortality and compliance. It is in this regard that surface properties of biomaterial should be modified so as to decrease adhesion of microbes, with increase antimicrobial properties and other beneficial properties. Surface properties which affects the function of biomaterials are mainly surface topology, surface charge etc [1]. Surface can be modified by coating, oxidation by low temperature plasma, surfactant addition for anti-static. Blending also uses for improving tensile properties and to provide a stronger structural component for separation media that supports the active polymer. Commonly used surface modification techniques include coating, oxidation by low-temperature plasma for better printing and adhesion and surfactant addition for anti-static.

Polyethylene glycol (PEG) is a water soluble polymer with many molecular weights that exhibits useful properties such as protein resistance, low toxicity and immunogenicity. PEG shows good solubility in water, biocompatibility, and drug carrier. PEG can be copolymerised with any other polymer for improving its properties.

Chitosan is the N-deacetylated derivative of chitin, a cationic polysaccharide composed of glucosamine and N-acetylglucosamine residues with 1, 4-b-linkage [5]. Chitin polysaccharide is one of the most abundant found in nature. PEG and chitosan both have excellent properties so they used in combination for increase properties like mechanical and biocompatibility. Antimicrobial properties as well as thermal and mechanical properties examined for performance testing.

II. MATERIALS AND METHOD

Chitosan purchased from sigma, polyethylene glycol from hi media, acetic acid, gluteraldehyde, nutrient agar.

2.1 Sample preparation

Pure polyethylene glycol:

Pure polyethylene glycol prepared for coating by dissolving 400 mg polyethylene glycol in to 5 ml distilled water. Then stir the solution over night for proper mixing. Coat this sample on a glass slab

Crosslinked polyethylene glycol:

5 ml polyethylene glycol was dissolved in 5 ml distilled water. After proper mixing add s µl gluteraldehyde for crosslinking. Stir the solution over night after that coat it on glass.

Polyethylene glycol and chitosan conjugate:

1 g of chitosan was dissolved in 100 ml, 2% acetic acid solution after that add 8 ml distilled water. Stir it till clear solution obtained. Then add 2 ml polyethylene glycol and stir for overnight.
III. RESULT AND DISCUSSION

3.1. Antimicrobial testing:
Antimicrobial testing has been done by agar diffusion method. Sample prepared for coating showed antimicrobial properties against E.coli. A clear zone of inhibition showed antimicrobial properties.

Fig. 1. (a) Antimicrobial properties of pure polyethylene glycol, (b) crosslinked polyethylene glycol, (c) Polyethylene glycol & chitosan conjugate against E.coli

Zone of inhibition (cm) of polyethylene glycol & chitosan conjugate has large diameter than two other samples, which showed strong antimicrobial properties.

Fig.2. Comparison of zone of inhibition of coating samples

3.2. Thermal analysis:
DSC curve showed glass transition temperature of pure polyethylene glycol which is 65˚c. Crosslinked polyethylene glycol has not any sharp peak due to moisture content, crosslinker [5]. Polyethylene glycol & chitosan have melting point 65˚c.

Fig.3. (a) DSC curve of pure polyethylene glycol, (b) Crosslinked polyethylene glycol, (c) polyethylene glycol & chitosan conjugate
3.3. Surface morphology:

SEM images shows in figures which shows surface morphology of pure polyethylene glycol, crosslinked polyethylene glycol, Polyethylene glycol & chitosan.

![SEM images of pure polyethylene glycol, Crosslinked polyethylene glycol, Polyethylene glycol & chitosan conjugate](image)

Polyethylene glycol & chitosan have smoother surface than other two samples. It results strong antimicrobial properties.

3.4. FTIR analysis:

FTIR analysis represents adsorption peaks depend on the frequency of vibration of bonds of atom of that material. Size of peak denote amount of materials. Moon-Sung Kang et.al, 2006 explained that FTIR spectra of neat PEG and PEG crosslinked with GA [2]. With increasing the GA content, the O–H stretching vibration band at near 3387 cm$^{-1}$ was decreased along with the band shift to a higher wave number at 3470–3432 cm$^{-1}$. This result suggests that the hydrogen bonding between –OH groups of PEG becomes weaker in crosslinked PEG than in neat PEG.

![FTIR curve of pure polyethylene glycol, Crosslinked polyethylene glycol, Polyethylene glycol & chitosan conjugate](image)

Table 1 wave number of FTIR analysis

<table>
<thead>
<tr>
<th>Bonding</th>
<th>Reference number</th>
<th>Wave number</th>
<th>Wave number (cm$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O–H bonding</td>
<td>3387</td>
<td>3470</td>
<td></td>
</tr>
<tr>
<td>C=O-C bonding</td>
<td>1298</td>
<td>1250.4119</td>
<td></td>
</tr>
<tr>
<td>C=O bonding</td>
<td>1724</td>
<td>1735.67</td>
<td></td>
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</tbody>
</table>

The appearance of the aldehyde C=O bond at 1735.67 cm$^{-1}$ with an addition of GA indicates that the aldehyde groups of GA do not completely react with –OH groups of PEG (i.e. non-functional reaction), and the unreacted aldehyde groups are also available for the coordination with other group [17].

In fig.6 most of the characteristic peaks of PEG (1467, 1280, 1089, 962 and 842 cm$^{-1}$) which is almost same to the polyethylene glycol & chitosan. Since no appearance of new peaks or disappearance of peaks of individual components, it means that there was no chemical reaction between chitosan and PEG.
3.5 Mechanical testing:

Crosslinked polyethylene glycol shows are good mechanical properties as compared to polyethylene glycol and chitosan conjugate film. When force is applied at stretched up breaks point after that film break this shows the breaks load of crosslinked polyethylene glycol. The mechanical properties of chitosan conjugate film are not good as compared to crosslinked polyethylene glycol film. When force is applied at stretched up breaks point after that film break this shows the breaks load.

The load bearing capacity depended on molecular weight of PEG [19]. The breaking point of PEG-PEG hydrogel was 10.5 kg and our result is 12.5 kg. So by comparative study it concluded that crosslinked polyethylene glycol film have high weight bearing capacity. For crosslinked polyethylene glycol it is 12.5 kg. Crosslinked polyethylene glycol has 4.4 % of elongation. Crosslinked polyethylene glycol has higher hardness as compared to polyethylene glycol & chitosan. Polyethylene glycol & chitosan conjugate sample shows not much good mechanical properties. Its mechanical strength is poor. The mechanical properties of chitosan and polyethylene glycol film can be improved by addition of crosslinker. Film of this conjugate is very soft that why it’s not easy to done tensile testing on it.

### Table 2 Thickness of coating films

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crosslinked polyethylene glycol</td>
<td>0.217mm, 0.302mm</td>
</tr>
<tr>
<td>Polyethylene glycol &amp; chitosan</td>
<td>0.058mm, 0.060mm</td>
</tr>
</tbody>
</table>

3.5.2. Hardness

Hardness of film is measured by Shore Hardness instrument. Hardness of crosslinked polyethylene glycol film is 75 - 82 shore.

3.5.3. Breaking load

Breaking load is the load at which film breaks. It’s measured 12.5kg.

3.5.4. % Elongation

Value of % elongation for polyethylene glycol film is 4.4%

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**IV. CONCLUSION & FUTURE WORK**

Chitosan and polyethylene glycol film is not mechanically strong. Mechanical properties of crosslinked polyethylene glycol are good as compared to other two samples. The mechanical properties of chitosan conjugate film are not good as compared to crosslinked polyethylene glycol film. For crosslinked polyethylene glycol it is 12.5 kg. Crosslinked polyethylene glycol has 4.4 % of elongation. Crosslinked PEG has higher hardness as compared to polyethylene glycol & chitosan. Greater the amount of PEG more will be the strength of film. Surface morphology of all three samples it concluded that pure polyethylene is rough in surface as compared to crosslinked polyethylene glycol & polyethylene glycol & chitosan conjugate. SEM images showed that polyethylene glycol- chitosan surface was smoother as compared to pure PEG & crosslinked PEG.

FTIR result showed that proper crosslinking of polyethylene glycol. FTIR results showed for polyethylene glycol 3422.00 cm⁻¹(O-H bond). DSC result of pure polyethylene glycol showed a high glass transition temperature than crosslinked polyethylene glycol & polyethylene glycol & chitosan conjugate. The different molecular weights of PEG had no significant influence on the mechanical properties and biocompatibility, perhaps because the molecular weight differences were not enough to significantly affect the material properties. DSC curve for pure polyethylene glycol showed sharp peak which show purity of sample but crosslinked polyethylene glycol and polyethylene glycol & chitosan have broad peaks.

Comparative study of different concentration of polyethylene with conjugate to chitosan can be use for blending. Use lysozyme in polyethylene glycol for enhancing antimicrobial activity. Copolymers can be use as coating material. Use of Dextran in place of polyethylene glycol because it has high-density surface immobilization of biologically active molecules to low protein-binding surface coatings is desired. Mechanical properties of chitosan and polyethylene glycol can be increase by using crosslinker.

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