THE GREEN SYNTHESIS OF CHITOSAN BASED SUPER ABSORBENT POLYMER TO ENHANCE WATER HOLDING CAPACITY OF SOIL

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ABSTRACT

Super Absorbent Polymers (SAP) are cross-linked hydrophilic polymers having three-dimensional network, which absorb and retain huge quantities of water than their original mass and volume, and release during the dry conditions. Super absorbent polymers are used as soil conditioner in agriculture for improving soil water holding capacity. The conventional hydrogels available in the market are acrylate-based products, which are not biodegradable and hazardous in nature. This research was attempted to synthesize superabsorbent polymers from renewable sources along with biocompatible cross linkers to make it completely biodegradable and biocompatible for agricultural use. In this current study, hydrogels were synthesized using chitosan cross-linked with polyethylene glycol, citric acid and oxalic acid. Chitosan based hydrogels were characterized for moisture content, equilibrium water absorbency, swelling ability in acidic (pH=4) and alkaline (pH=10) conditions. The surface morphology and cross-linking of hydrogel was characterized through electron microscopy and Fourier transform infrared spectroscopy respectively. The results showed that equilibrium water absorbency of chitosan cross-linked with polyethylene glycol was 3851 per cent of hydrogel followed by hydrogel with oxalic acid retained 3130 per cent. Similar trend of swelling capacity was observed in acidic and alkaline pH. Soil water holding capacity amended with hydrogels was determined through Keen-Rackzowski box, which revealed that soils amended with chitosan – graft- oxalic acid @ 0.1 per cent exhibited higher water holding capacity even after drying for 7h in oven at 70°C.

KEYWORDS: Chitosan, Super Absorbent Polymer, Swelling Capacity & Soil Water Holding Capacity

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1. INTRODUCTION

India accounts 4 per cent of the world fresh water supporting 17 per cent of the world’s population. India has significant spatial mismatches of the population intensity and water resources. Less water is available in places where more people live and much of the food is grown. It is estimated that nine river basins habituating with 75 per cent of the Indian population, will be physically water-scarce by 2050 (Amarasingh et al. (2007). The frequency of drought year’s occurrence in India is expected to increase between 2020 and 2049. Hence, water saving technologies in agriculture is crucial for improving agricultural productivity. Super absorbent polymers are hydrophilic macro molecules with three-dimensional structure in dispersion medium attaining few tens, hundreds

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or thousand times of body mass volume on absorption of water without dissolving in the dispersal medium (Schacht, 2004). The application of super absorbent polymers as soil conditioner is practiced in crop production for improving soil water holding capacity and releasing water to the crop during drought conditions (Vundavalli. R. et al., 2015).

The network structures in hydrogel are achieved through physical or chemical cross-linking of hydrophilic polymer chains (Ekebafe. L. et al., 2015). Hydrogels swell water to an equilibrium value and maintain the physical dimension structure (Zohuriaan-Mehr and Kabiri, 2008). Different synthetic, semi-synthetic (modified natural) and natural polymer are used to develop super adsorbent polymers. The use of hydrogels increase the water-holding capacity of sandy soil in arid regions (Bakass et al., 2002). The swelling ability of hydrogels depends on the functional acidic or basic groups at the polymer backbone, while the water holding capacity of soil amended with hydrogels depend on soil matrix and bead size of hydrogel (Bhardwaj et al., 2007). The rate of water evaporation in soil–hydrogel mixtures are slower because of reduced hydraulic gradient that drives water for evaporation (Yu et al., 2012). Generally, acrylic based super absorbent polymers are used extensively in the agriculture, which are not biogradable and biocompatible which limited the adoption of super absorbent polymers as soil conditioner in agriculture (Barros et al., 2017). Chitosan is a co-polymer of biological origin with varying amounts of β (1 4) linked residues of N-acetyl-2 amino-2-deoxy-D-glucose (glucosamine) and 2-amino-2-deoxy-D-glucose (N-acetyl-glucosamine) residues, which is second most abundant polymer after cellulose. Chitosan is a deacetylated form of chitin, which is the major structural component of crustaceans and insects. The presence of both amine and hydroxyl functional groups in the chitosan has high potential for modifications facilitating various applications (Kumar, 2000). The cationic character of chitosan favors the formation of gel particles through electrostatic interactions with cross-linking agents. Chitosan hydrogel holds the water tightly in the polymer matrix while the surface of the matrix contains free water (Rohindra, et al., 2004). The poly ethylene glycol, oxalic acid and citric acids were shown to work as cross-linking agents with biocompatible nature. Oxalic acid is a dicarboxylic acid (COOH–COOH) with a low dissociation constant capable of forming weak acid–base interactions in polymer electrolytes which facilitates protonation with chitosan (Missan et al., 2006). Similarly, the presence of hydroxyl function group in polyethylene glycol and carboxylic group in citric acid favors the ionic interaction with amine groups of chitosan. The current research was aimed on synthesizing the chitosan based superabsorbent polymer to use as soil conditioner for improving soil water holding capacity. In the present study, chitosan was cross-linked with different amounts of polyethylene glycol/oxalic acid/citric acid for the synthesis of hydrogels. The swelling behavior of the gels in aqueous media was characterized for the water uptake in the hydrogels

2. MATERIALS AND METHODS

2.1. Materials

Chitosan (Mn=190–310 kDa) ≥75% deacetylated, Glacial acetic acid, Polyethylene glycol(PEG 6000) were purchased from Sigma-Aldrich Chemicals PVT Ltd and used as such for the experiment. Citric acid and oxalic acid used in this study were obtained from Astron chemicals (India) Limited. Deionized water was used in dissolution of chitosan for graft polymerization and swelling experiments.

2.2. Preparation of Chitosan-Graft- Polyethylene Glycol /Oxalic Acid /Citric Acid Based Super Absorbent Polymer

Chitosan-graft-polyethylene glycol/oxalic acid/citric acid super absorbent polymer formulation was synthesized using ionic-linking method. Chitosan(3%w/v) was dissolved using glacial acetic acid (1.4% v/v) used magnetic stirred
at 100 rpm for one hour to dissolve completely. The calculated quantity of polyethylene glycol/oxalic acid/citric acid were added to the chitosan solution, where aqueous phase of chitosan turns into gel phase. The gel was transferred to conventional laboratory oven for drying at 70°C for 3 to 4 h. The product was stored at ambient temperature for further analysis and characterization (Figure 1)

2.3. Characterization

2.3.1. Equilibrium Swelling Ratio of Hydrogel

Swelling behavior of chitosan-based super absorbent polymer was assessed by immersing known quantity of hydrogels in deionized water at room temperature to reach the equilibrium state. The swollen gels were taken out, and wiped superficially with filter paper to remove surface bound water and weighed. The equilibrium state of hydrogels reached where there is no difference in the mass of swollen hydrogels. Equilibrium degree of swelling and equilibrium water content of hydrogels were calculated with following formulae

\[
\text{Equilibrium degree of swelling} \% = \frac{W_1 - W_2}{W_2} \times 100
\]

\[
\text{Equilibrium water content} \% = \frac{W_1 - W_2}{W_1} \times 100
\]

where \(W_1\) is the weight of swollen gel and \(W_2\) is the weight of dry gel.

2.3.2. Water Absorption Ratios of Hydrogels under Various pH and Saline Conditions

Swelling ability of hydrogels estimated as measure of water uptake was estimated at various saline and pH levels. The absorption capacity of the synthesized hydrogels was measured in buffer solutions at pH 4 and pH 10 using tea bag method. A known quantity of hydrogel taken into a tea bag was immersed in an excess amount of water at room temperature. After a certain period, the bag was removed from the solution, where the swollen gel was wiped for removing excess water with filter paper. The swelling ratio was estimated using the formula as mentioned for estimating equilibrium degree of swelling.

2.3.3. Soil Water Retention Measurements

An estimated quantity of soil after passing through 2-mm sieve was used for water retention studies. Hydrogels (0.1% w/w) were mixed with soil and transferred to Keen-Rackzowski box (Keen box), where the bottom of the box was covered with Whatman paper No.1 and No. 44. Then the soil was filled in the box forging with repeated tapings to ensure uniform packing of soil inside the box. The keen box with air-dry soil weighing 50g was placed inside the plastic tray with water, where the water level was maintained at just one cm above the base of the keen box (Figure 2). The tray was covered with plastic film to prevent the evaporation of water from the soil surface. The keen box was kept at room temperature for 12 h to reach equilibrium state. Then the box was carefully removed and allowed to drain free water from the box and weighed. The keen box was transferred to an oven for continuous drying at 70°C for 7 hours, and samples were weighed at hourly interval to plot the magnitude of dryness over the time. The degree of water absorbed by hydrogel soil mixture was calculated and expressed in weight percentage (Yu et al., 2017)

\[
\text{Water absorbed} \% = \frac{L}{M} \times 100
\]

where \(L\) is the weight of the water in soil hydrogel mixture at a given drying time (calculated as the difference between the weight of the wet soil hydrogel mixture and weight of the dry soil and dry hydrogel (0.1g); \(M\) is the weight of
2.3.4. Rheological Studies

The viscosity of chitosan based hydrogels were assessed using rotating Rheometer (Model DVT2, M/s Brookfield Engineering Laboratories, USA) operated at 0.1 rpm for 2 min with spindle (CP 41). Sample size of 5 ml was used for the measurements.

2.3.5 Scanning Electron Microscopy

Scanning Electron Microscopy imaging of the hydrogels was performed to assess the external structure of the polymer before and after cross linking. Dried hydrogels were placed in the carbon-coated stubs, and sputter coated with 10nm gold using Emitech, SC 7620 Basic Magnetron Sputter Coater. The sputter-coated hydrogels were imaged with scanning electron microscope (Quanta 250, FEI, Netherlands) at 10KV with spot size of 3.5

2.3.6. Fourier Transfer-Infrared Spectroscopy (FTIR)

FTIR is a powerful tool to identify chemical bonds in a molecule of specific sample, often referred as molecular "fingerprint". The FTIR analysis was carried out to confirm the cross linking of polymers. The small portion of dried hydrogel sample were placed in the sample contact area in the spectrometer (JASCO FT-IR 6800 Spectrometer) and scanned at ATR mode using ATR PRO ONE. Samples were scanned with a resolution of 4cm⁻¹ and 64 times with 400 - 4000 cm⁻¹

3. RESULTS AND DISCUSSIONS

3.1. Equilibrium Swelling Ratio of Hydrogel

Water molecules diffuse into the hydrogel when immersed in water, until it reaches equilibrium state. The time taken for reaching equilibrium state between chitosan – graft – polyethylene glycol/oxalic acid/citric acid hydrogels ranged from 13-15 minutes. The equilibrium degree of swelling and equilibrium water content of hydrogels are presented in Figure 3 and Figure 4 respectively. Equilibrium degree of swelling was maximum in chitosan – graft – polyethylene glycol hydrogel, which was about 3851 per cent, whereas the equilibrium degree of swelling of chitosan – graft – oxalic acid hydrogel was 3131 per cent. Chitosan – graft – citric acid hydrogel recorded the least equilibrium degree of swelling (887 per cent). Equilibrium water content indicates the actual water uptake by hydrogels. The highest equilibrium content was observed in chitosan – graft – polyethylene glycol hydrogel (126 per cent), while it was followed by chitosan – graft – oxalic acid hydrogel (109 per cent). The equilibrium degree of swelling and equilibrium water content of hydrogels are presented in Figure 3 and Figure 4 respectively. Water uptake of hydrogels indicate the entry of water molecules into preformed pores between the polymers chains through cross-linking. The density of hydrogels and ionic strength of cross-linkers determines the accessibility of water molecules to hydrophilic portion of hydrogels and strength of hydrogels (the ability of hydrogel to maintain homogeneous structure). The denser the hydrogels result in the decreasing the entry of water molecules into the hydrogel.

3.2. Water Uptake of Hydrogel under Saline and Varied pH Conditions

External factors such as pH, temperature and salt concentration of aqueous medium affects the swelling behavior of super absorbent polymers. The ions present in the aqueous medium affect the water uptake of hydrogels significantly, which is explained by the Donnan equilibrium theory. The electrostatic interactions between the aqueous medium and...
hydrogel is driven by the difference between the osmotic pressure of the free mobile ions in the polymer and the saline solutions. Hence, the osmotic potential difference due to the concentration ions between medium and hydrogel determines the swelling behavior of hydrogels. Wider the concentration difference between the aqueous medium and hydrogel reduces the hydrogel volume in turn decreasing the swelling capacity. Salinity levels (0.1 and 0.9 per cent) affect the water uptake of chitosan-based hydrogels (Figure 5 and 6.) The results revealed that water uptake of chitosan – graft – oxalic acid hydrogel absorbed 15.6 and 12.0 g g\(^{-1}\) of water @ 0.1 and 0.9 per cent saline solutions respectively, while the chitosan – graft – polyethylene glycol hydrogel recorded 12.4 and 10.2 g g\(^{-1}\) under salinity levels of 0.1 and 0.9 per cent respectively. The water uptake was poor in chitosan – graft – citric acid hydrogel at both salinity levels. Swelling ratio of hydrogels decreases with increase in salinity of water which may be attributed to the charges of cations (Na\(^+\)) present in the water. The decrease in swelling of chitosan based hydrogels may be due to the formation of ionic bonds between cation (Na\(^+\)) of water and OH groups of chitosan in the hydrogel, where with increasing the charge of cation, more ionic bonds may be formed which decreases the number of the hydrophilic groups in the hydrogels. Similar to the effect of temperature on hydrogel, swelling behavior of chitosan-based hydrogels varied with pH conditions (Figure 7). Extremes of pH in water medium reduces the absorbing ability of hydrogels, which may be due to the charge overlap effect (Wan et al., 2016). The swelling of chitosan graft hydrogels increases with increasing gradient of pH in the medium except for the chitosan – graft – citric acid hydrogel, where it was recorded that there was a dip in swelling near the neutral pH. The reason for more swelling in higher pH may be due to the deprotonation in the polymer chains.

3.3 Soil Water Retention Studies

Keen-Rackzowski box experiments were carried out to estimate soil water holding capacity and water retention characteristics of chitosan graft polymers. Keen box studies also revealed the water absorption and release from soil–chitosan hydrogel mixtures during 7 hours of drying (Figure 8). Water retention capacity in chitosan – graft – oxalic acid hydrogel amended soil was higher (17.1 per cent) after 7 h of drying in oven at 70°C, while it was 11.4 % for soils amended with chitosan – graft – polyethylene glycol hydrogel. The highest water holding capacity in chitosan – graft – oxalic acid hydrogel after 7 h of drying may be an outcome of the higher stability of hydrogels and ionic strength of cross linker. The ionic strength of oxalic acid was higher (1.328 mol kg\(^{-1}\)) in contrast to polyethylene glycol (0.332 mol kg\(^{-1}\)). The ionic strength of citric acid used in the study was 0.331 mol kg\(^{-1}\). The water retention studies revealed that water release from chitosan – graft – oxalic acid hydrogel amended soil was prolonged during the drying compared to that of other soils amended hydrogels. The ionic strength of cross linker is significant for maintaining homogeneity of hydrogel subjected to drying.

3.4. Characterization of Chitosan Graft Polymers

Rheological behavior of hydrogels is an important factor determining the optimum flow and the proper crosslinking, which are based upon the concentration of polymer and the cross linking agents. The results on viscosity showed that there is a decrease in viscosity of chitosan cross-linked hydrogels in comparison compared to that of the non-cross-linked chitosan. The highest viscosity (1117 cP) was observed in chitosan – graft – polyethylene glycol followed by chitosan – graft – oxalic acid (759.8 cP). The viscosity of chitosan – graft – citric acid was 633cP, while the viscosity of chitosan (1.4%) alone was 4870 cP without cross linking. The arm length of polyethylene glycol contributes for higher viscosity of hydrogel in spite of its lower ionic strength. Generally, higher the ionic strength of crosslinking agents increases the viscosity of hydrogels because of the cross linking efficiency with polymers. The viscosity of chitosan – graft
- citric hydrogels was lower because of less ionic strength of citric acid. The lower ionic strength of citric acid resulted in poor cross linking efficiency. The presence of citric acid molecules embedded in chitosan polymer chain becomes more repulsive in nature among themselves that lead to stretch the polymer matrix and lower the viscosity of polymer (Shivani et al., 2013).

Scanning electron micrography of a cross-section and a surface of chitosan graft hydrogels was carried out after sputter coating with gold, which was depicted in Figure 9a – 9d. The electron microscopy images showed the presence of porous structure distributed uniformly in cross-linked hydrogels, whereas no pores were observed over the surface of non-cross linked polymer. The pores are sponge-like shaped with spherical openings that are highly interconnected. The porous microstructure in the hydrogel assures the capillary effect of water uptake. It was also recorded that the surface of cross-linked hydrogels had distribution of small pores throughout the surface of hydrogels.

Infrared spectra of chitosan and polymerized hydrogels with polyethylene glycol, oxalic acid and citric acid was presented in Figure10a to 10c. The infrared spectra of chitosan – graft – polymers showed that there was broadening of peak of -OH group corresponding to the region of 3600 to 3100 cm\(^{-1}\) compared to that of non-cross-linked chitosan. Similarly, overlapping of -NH vibrations corresponding at 2970 cm\(^{-1}\) with -OH stretching was observed in the cross-linked hydrogels. The broadening of -OH peak confirms the crosslinking of polymers. Relatively less broadening of -OH stretching was observed in chitosan- graft – polyethylene glycol hydrogels. The absorbance peak at 2926 cm\(^{-1}\) and 1404 cm\(^{-1}\) belongs to asymmetric stretching and bending of C – H of chitosan. Sp\(^3\)hybridization of carbon with nitrogen atom present in amine group and aliphatic nitro compounds of chitosan was reflected through the peak at 1016 cm\(^{-1}\); 1382 cm\(^{-1}\); 1593 cm\(^{-1}\) and 1651 cm\(^{-1}\). The absorbance peaks at 1300 cm\(^{-1}\); 1263 cm\(^{-1}\); 1063 cm\(^{-1}\) and 3627 cm\(^{-1}\) in infrared spectra of chitosan represent C – N and C – O – C stretching and -OH stretching. The shift of amine peak from 1651 cm\(^{-1}\) to 1648 cm\(^{-1}\) represent the interaction of amine group in chitosan with -OH group of polyethylene glycol confirming the cross-linking in chitosan polymer chain. The absorbance peak at 1593 cm\(^{-1}\) in chitosan corresponding to primary amine group was absent in chitosan – graft – polyethylene glycol hydrogels indicating the distortion of -NH\(_2\) group during crosslinking (Figure 10a). The shift of peak from 3153 cm\(^{-1}\) to 3175 cm\(^{-1}\) was observed in chitosan – graft – oxalic acid hydrogel due to the interaction of - OH group of chitosan with that of the disassociated hydrogen atom of oxalic acid. The strong broad peak at 1534 cm\(^{-1}\) was due to the presence of -COO\(^-\) group indicating the protonation of NH\(_2\) in chitosan by the dissociated H\(^+\) from oxalic acid (Figure 10b). The appearance of new peak at 1560 cm\(^{-1}\) corresponding to the presence of amide II bond was observed in chitosan – graft – citric acid. The peak at 1380 cm\(^{-1}\) in the hydrogel might represent vibration in COO\(^-\) ions confirming the crosslinking of citric acid in the chitosan polymer(Figure 10c). Shift and appearance of new peaks in cross-linked chitosan polymers confirms the polymerization and crosslinking processes, whereas broadening peak at OH stretching in hydrogels confirms the addition of water molecules

4. CONCLUSIONS

Super absorbent polymers are interesting area of research in agriculture which are used as soil conditioner and template for improving the soil water holding capacity besides the controlled release of agricultural inputs. Now-a-days, green chemistry is the principle of chemical synthesis due to awareness on the enviromental concerns to protect the services of ecosystem. Hence, it was attempted to synthesize chitosan based super absorbent polymers crosslinking with biocompatible crosslinkers such as polyethylene glycol, oxalic acid and citric acid under low quantity of energy consuming process. The results on water uptake studies showed that chitosan – graft – polyethylene glycol hydrogel absorbed higher
The quantity of water when immersed in free water, while chitosan–graft–oxalic acid hydrogel retained more water under drying conditions in Keen box experiments. The variation in swelling behaviour of hydrogel under free water and water deficit conditions are due to the nature of interactions during crosslinking and the ionic strength of the crosslinkers. The performance of chitosan-graft hydrogels as a template for the delivery of agricultural chemicals may be ascertained for its application efficiency. Similarly, the crosslinkers with bi-functional groups and concentration of polymers may be explored during polymerization and crosslinking processes for improving the swelling capacity of hydrogels.

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APPENDIX

Figure 1: Appearance of Chitosan – Graft – Polyethylene Glycol Hydrogel after Reaching Equilibrium State

Figure 2: Keen-Rackzowski box Experiment to Determine Soil Water Retention Capacity in Soils Amended with Hydrogel and Control (without Amending Hydrogel)

A: Control; B: Soil amended with hydrogel crosslinked oxalic acid; C: Soil amended with hydrogel crosslinked with citric acid and D: Soil amended with hydrogel crosslinked with poly ethylene glycol

Figure 3: Equilibrium Swelling Degree of Chitosan - Graft – Polyethylene Glycol/ Oxalic Acid/ Citric Acid
Figure 4: Equilibrium Water Content of Chitosan - Graft - Polyethylene Glycol/ Oxalic Acid/ Citric Acid

Figure 5: Water Uptake of Chitosan Graft Polyethylene Glycol/ Oxalic Acid/ Citric Acid in Salinity Level (0.1 per cent of NaCl)

Figure 6: Water Uptake of Chitosan Graft Polyethylene Glycol/ Oxalic Acid/ Citric Acid in Salinity Level (0.9 per cent of NaCl)
Figure 7: Water Uptake of Chitosan Graft Polyethylene Glycol/ Oxalic Acid/ Citric Acid in Varied pH Levels (pH 4, 7 and 10)

Figure 8: Water Retention Studies of Chitosan Graft Polyethylene Glycol/ Oxalic Acid/ Citric Acid using Keen box Experiments

Figure 9: Scanning Electron Microscopy Images (a) Non Crosslinked Chitosan; (b) Chitosan - Graft - Polyethylene Glycol Hydrogel; (c) Chitosan - Graft - Oxalic Acid Hydrogel and (d) Chitosan - Graft - Citric Acid Hydrogel
Figure 10: Infrared Spectra; (a) Chitosan - Graft - Polyethylene Glycol Hydrogel; (b) Chitosan - Graft - Oxalic Acid Hydrogel and (c) Chitosan - Graft - Citric Acid Hydrogel
