Morphological and Swelling Potential Evaluation of *Moringa oleifera* Gum/Poly(vinyl alcohol) Hydrogels as a Superabsorbent

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ABSTRACT: Synergistic use of superabsorbent hydrogels has vital contribution to the daily life. This work gives an account of a facile approach to synthesize superabsorbent hydrogels based on *Moringa oleifera* gum and polyvinyl alcohol cross-linked with borax. Fourier transform infrared, X-ray diffraction, and scanning electron microscopy were employed to characterize the structure, crystalinity, and surface morphologies of the samples. The cross-link density, swelling ratio, reswelling, water retention properties, and salt sensitivity of hydrogels were investigated. Reaction parameters for the hydrogel synthesis were optimized on the basis of water absorbency, and the reaction condition of greater water absorbency after 12 h at room temperature was taken as an ideal condition. Optimum conditions were obtained as [poly(vinyl alcohol) PVA] = 10% (w/v) and [borax] = 1.05 × 10⁻³ mol/L. Under the optimized conditions, the maximum swelling ratio of MOG/PVA hydrogel reached 1163 g/g in deionized water and 290 g/g in 0.9 wt % NaCl solution. Furthermore, all hydrogels exhibited salt sensitivity and excellent water retention capacity under the high temperature state and displayed smart swelling behaviors in physiological saline solutions. The water absorbency, reusability, and salt sensitivity of the hydrogels give these smart polymer wide promising applications.

1. INTRODUCTION

Superabsorbent hydrogels (SHs) are three-dimensional networks of linear (or branched) hydrophilic polymers interconnected by chemical and/or physical cross-linking. They can absorb and retain large quantities [swelling ratio (SR) > 100] of liquids, blood solutions, and bodily fluids. SHs are broadly used in agriculture, biomedical applications, personal care products, and even in sewage treatments. Moreover, on the basis of their functional groups including −COOH, −OH, −CONH₂, −SO₃H, and their cross-linking nature, SHs may perform by various stimuli factors such as osmotic pressure, pH, and temperature, which results in a gradual or sudden variation in the SR.

Commonly, water-absorbing gels experience swelling in various phases. Initially, the gel outside comes in contact with water molecules and become wetted through hydrophilic groups present on the gel surface. Second, the water molecules enter in polymer matrices through micropores, which leads in the expansion of gel molecular volume. Lastly, the osmotic pressure of water and the binding force of the molecular matrices reached to equilibrium, as a result, the most extreme water absorbency is achieved. However, the polymer hydrophilic groups tie salt constituent particles effectively by a mechanism similar to complexation. Therefore, the swelling capacity decreases abruptly in aqueous fluids, such as blood, urine, and soil solutions. To fulfill these gaps, a simple tactic applied to synthesize superabsorbent materials depends upon natural polysaccharide such as natural gums, cellulose, chitosan, and starch, which might rise their biocompatibility, biodegradability, and swelling capacity.

*Moringa oleifera* (family: Moringaceae) is a wild plant that grows in many countries of tropics and subtropics. The stem of the *M. oleifera* tree exudes a gum commonly known as *M. oleifera* gum (MOG), which is initially white in color but changes to brownish black and reddish brown on exposure. It gives a highly tacky solution when dipped in water. MOG is composed of D-galactose, D-mannose, D-glucoronic acid, D-xylose, L-arabinose, and L-rhamnose. All the parts of *M. oleifera* are nontoxic and beneficial for human health and contain minerals, nutrients, and various antioxidants. In India, MOG is usually known as sajna gum.

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Polymer like poly(vinyl alcohol) (PVA) has fair reputation as a biocompatible and nontoxic linear biopolymer.\textsuperscript{21} We selected PVA as the other component because of its fair adhesive properties and water solubility.\textsuperscript{22} The aim of this work was to develop natural gum-based SH with better swelling, physicochemical, and reswelling properties that can vie with synthetic counterparts concerning to the environmental problems. The hydrogel was synthesized using biodegradable and cheap materials: MOG and PVA. The synthesis procedure is aqueous-based, catalyst-free, and environmentally friendly. Here, we described its advantages, procedure, and swelling properties to evaluate its promising application.

2. RESULTS AND DISCUSSION

2.1. Fourier Transform Infrared Spectra. Fourier transform infrared (FTIR) analysis (Figure 1) of MOG/PVA hydrogel displayed a strong absorption band in the region 3401 cm\textsuperscript{-1}, which can be assigned to stretch vibrations of O–H. The band in the region at 2906 cm\textsuperscript{-1} is because of C–H symmetric stretching vibrations. Further a wavenumber at about 1663 cm\textsuperscript{-1} represents hydroxyl bending mode. The characteristic absorption bands at 1163 and 1457 cm\textsuperscript{-1} correspond to C–O–C stretching and CH\textsubscript{2} bending vibrations, respectively. In case of cross-linked hydrogel, the strong absorption band of MOG\textsuperscript{17} at 3403 cm\textsuperscript{-1} and PVA\textsuperscript{23} at 3400 cm\textsuperscript{-1} was less pronounced and slightly moves to longer wavelength. The appearance of bands around 1429 and 1343 cm\textsuperscript{-1} is because of asymmetric stretching of B–O–C, and also, a wavenumber around 1661 cm\textsuperscript{-1} corresponding to H–OH bending has weakened, and a wavenumber around 837 cm\textsuperscript{-1} corresponding to C–H bending has almost disappeared, which provides evidence that the cross-linking between hydroxyl groups of MOG and PVA by borax.\textsuperscript{24} 

2.2. X-ray Diffraction Studies. The X-ray diffraction (XRD) pattern (Figure 2) of MOG exhibits a broad peak around 2θ = 10–28°, which corresponds to the amorphous nature of pure MOG.\textsuperscript{17} The PVA showed two typical peaks\textsuperscript{25} at 2θ of 19.4 and 41°. In case of MOG/PVA hydrogel, the intensity of diffraction peaks at 2θ = 21.9° decreases with slight change in position, suggesting decomposition of semicrystalline and amorphous nature of PVA and MOG, respectively, which indicates that MOG and PVA were chemically cross-linked.

2.3. Scanning Electron Microscopy. Scanning electron microscopy (SEM) images of MOG, PVA, and borax cross-linked MOG/PVA hydrogel are presented in Figure 3a–c, respectively. The MOG exhibited a smooth and compact surface, as shown in Figure 3a. PVA has a smoother and sheetlike morphology, as shown in Figure 3b, whereas the cross-linked hydrogel exhibited a fractured and scaly surface with significant pore spaces, as shown in Figure 3c. These pores are the regions for water permeation and interaction sites of external stimuli with the hydrophilic groups, which leads to increase in SR.

2.4. Swelling in Deionized Water. Figure 4 shows the SR of MOG/PVA hydrogels at different concentrations of PVA and borax at room temperature in deionized water. The water absorbency of hydrogels increases sharply during the first 120 min and afterward turned out to be slower until reaching a plateau. The swelling ability of hydrogels decreases with increasing cross-linker borax concentration, owing to the reason that the increase of cross-linking density makes polymer chains self-entangled, resulting in more compact structure. However, when the PVA content increases, the swelling of hydrogels first increased and then decreased, and the initial increase in swelling may be because of the increase in hydrophilicity and the formation of network of optimum pore size. Thereafter, further increase in PVA concentration has led to decrease in swelling of hydrogels, this may be because of the blocking of active sites on polymer chains, leading to decrease in the hydrophilic groups. The best result was obtained with SR 1163 (g/g), which was in agreement that the MOG/PVA hydrogels were superabsorbent.

2.5. Swelling in Various Saline Solutions. Figure 5 shows the equilibrium swelling capacity of MOG/PVA hydrogels in various saline solutions (D-glucose, urea, physiological saline water, and synthetic urine). The order of equilibrium swelling capacity for SHs in above solutions in the decreasing order was ranked as urea solution > D-glucose solution > physiological saline water > synthetic urine. The equilibrium swelling capacity of SHs in D-glucose solution was found to be slightly lower compared to the values measured in deionized water, whereas the equilibrium swelling capacity subsequently decreases in synthetic urine and in physical saline water. The shrinking behavior of hydrogels in synthetic urine and physiological saline water is because of the charge-
screening effect of the counterions (Na\(^+\), K\(^+\), Ca\(^{2+}\), and Mg\(^{2+}\)) resulting in the decline of anion–anion repulsions, which reduces the osmotic pressure between the internal hydrogel network and the external solution.\(^{26}\)

2.6. Water Retention Capacity. Figure 6 depicts the water retention capacity of MOG/PVA hydrogels. As can be seen from the curves, the water retention capacity decreases with increasing time. After dehydration, gel samples became plastic-like; moreover, water retention decreases with increasing borax amount. GEL 3 loses water rapidly compared to GEL 2, and became highly dehydrated after 5 h, while water retention capacity of GEL 2 was reached to 31\% at this time interval. This may be because of the H-bonding interactions and van der Waals forces between water molecules and hydrogel matrices, which directly affect the water retention capacity of hydrogel.\(^{27}\)

Considering the above, MOG/PVA hydrogels have outstanding water retention capacity, which allows for a further application prospects in agriculture.

2.7. Reswelling Capability Studies. The reswelling capability of hydrogels in deionized water following five consecutive swelling/drying cycles is depicted in Figure 7. The enormous weight and larger pressure inside the hydrogel system are caused by absorbed water molecules, resulting in the breaking of polymer networks.\(^{28}\) Thus, the voids in the hydrogel porous structure collapses slowly, resulting in the reduced swelling capability. The equilibrium swelling capacity of bone-dried GEL 2 was 1035 g/g for cycle 1 and remained at 756 g/g for cycle 5, exhibiting swelling capability loss to 35\%. The swelling capability of GEL 4, GEL 1, GEL 5, and GEL 3 after five reswelling cycles decreased to 40, 49, 58, and 72\%, respectively. These results infer the reason of hydrogen-bonding interactions among MOG and PVA that arisen during the drying process.

2.8. Salt-Sensitivity Analysis. To examine the salt-sensitivity factor (\(f\)) of the MOG/PVA hydrogels, the swelling capacity at equilibrium (\(W_s\)) in various salt solutions (NaCl, KCl, NH\(_4\)Cl, CaCl\(_2\), and AlCl\(_3\)) at 0.15 mol/L concentration is given in Table 1. In NaCl, KCl, and NH\(_4\)Cl solutions, the equilibrium swelling capacity \(W_s\) of all hydrogels increased in the following ascending order: Na\(^+\) < K\(^+\) < NH\(_4\)\(^+\) (Table 1).
This may be because of the charge screening effect of these cations on the $W_s$ values increased in the same order. The equilibrium swelling capacity in NaCl, CaCl$_2$, and AlCl$_3$ decreases with the increasing of cationic charge (Na$^+$ < Ca$^{2+}$ < Al$^{3+}$) (Table 1), which might be elucidated by the Donnan equilibrium theory. According to Donnan, the shrinking and swelling behavior were ascribed to the decrease osmotic pressure difference between the interior hydrogel and the external solution.

2.9. Network Parameters. The gel content influences the cross-link density (CD) of the hydrogel network and it rely upon the composition of the reaction mixture. Higher swelling capacity is because of low CD of hydrogels, and at lower CD, the water permeation into the hydrogel matrix becomes easier, resulting in the increment in gel-swelling capacity. Table 2 shows with the increase in borax concentration, the CD of the forming gel gets increased while $M_s$ decreased, this may be because of the number of efficient cross-links per unit volume increases in the hydrogel network, leading to a reduction in pore size of the voids present in the network chains. CD of gel 2 is lower than gel 4 and gel 5 because of the presence of hydrophilic functional groups and adequate pore size. Moreover, the network parameters influence the hygroscopicity, strength, and integrity of hydrogels.

3. CONCLUSIONS

In this research work, a series of borax cross-linked MOG/PVA hydrogels were developed and optimized as the water conservation material in agriculture. The procedure for hydrogel synthesis is ecofriendly as it employs biodegradable, catalyst-free, and inexpensive materials. The SRs of MOG/PVA hydrogels changed dramatically from 276 to 1163 g/g, depending upon the composition of the reaction mixture (borax and PVA). The swelling and shrinking behavior of MOG/PVA hydrogels were influenced by inorganic salt aqueous solutions. The water retention test confirms the good water holding property of the MOG/PVA hydrogel. This biobased SH has great potential for large-scale commercial applications including agriculture and hygienic products.

4. EXPERIMENTAL SECTION

4.1. Materials. MOG (Nutramine Life Sciences, Delhi), polyvinyl alcohol (SD Fine Chemicals India, M_w 85,000–125,000, alcoholysis degree 98–99%), and borax (Qualigens Fine Chemicals) are used. Deionized water was used throughout the experimentation. The solvents and chemicals were used without any further purification.

4.2. Preparation of the MOG/PVA Hydrogel. For the preparation of MOG/PVA hydrogel, definite concentration of MOG (4% w/v) was hydrated and stirred at 200 rpm with a overhead stirrer for 6–8 h at room temperature. PVA solutions of different concentrations (8% (w/v), 10% (w/v), and 12% (w/v)) were prepared separately by dissolving required quantity of PVA in deionized water at 80 °C. The cross-linker borax was separately dissolved in the calculated amount in deionized water (Table 1) and was added to the reacting solution mixture and again stirred for 30 min and kept aside for another 4 h without stirring for proper cross-linking. To remove unreacted borax, the hydrogels were rinsed with deionized water and soaked overnight in acetone for dehydration. Dehydrated hydrogels were filtered through a nylon sieve and dried in an oven at 45 °C till constant weight was obtained. The procedure for the synthesis of MOG/PVA hydrogel is described in Figure 8a, whereas dried and swollen hydrogels are depicted in Figure 8c,d, respectively.

4.3. Characterization of the MOG/PVA Hydrogel. FTIR spectra of MOG, PVA, and MOG/PVA hydrogel were recorded on a FTIR spectrophotometer (model: Tensor 37 spectrophotometer), within the frequency range of 4000–500 cm$^{-1}$ by the KBr pellet method. XRD patterns of MOG, PVA, and MOG/PVA hydrogel were collected using an X-ray diffractometer (model: Bruker) with Cu Kα radiation (λ = 1.54 Å) at a voltage of 40 kV and a current of 40 mA in the 2θ range of 10–80°. The surface morphology of MOG, PVA, and MOG/PVA hydrogel was observed by SEM (model: S-4800 Hitachi, Japan). Before the analyses, a thin layer of gold was sputter-coated on the sample surface.

4.4. Swelling Measurements. A series of accurately weighed bone-dried hydrogel samples (0.1 ± 0.0001 g) was dipped entirely in deionized water or various saline solutions (urea solution: 50 g urea in 1000 mL deionized water; 0.9 wt % NaCl aqueous (saline) solution; D-glucose solution: 50 g D-glucose in 1000 mL deionized water; synthetic urine: 0.6 g

Table 1. Effects of Salt Type on Equilibrium Swelling Capacity ($W_s$) and Salt-Sensitivity Factor ($f$) of MOG/PVA Hydrogels

| salt     | $W_s$ | f   | $W_s$ | F   | $W_s$ | f   | $W_s$ | f   | $W_s$ | f   |
|----------|-------|-----|-------|-----|-------|-----|-------|-----|-------|-----|
| NaCl     | 175   | 0.827 | 213   | 0.816 | 83    | 0.843 | 201   | 0.821 | 99    | 0.836 |
| KCl      | 191   | 0.811 | 231   | 0.801 | 91    | 0.827 | 219   | 0.805 | 107   | 0.822 |
| NH$_4$Cl | 207   | 0.795 | 249   | 0.785 | 101   | 0.808 | 237   | 0.789 | 119   | 0.802 |
| CaCl$_2$ | 83    | 0.918 | 106   | 0.909 | 39    | 0.926 | 97    | 0.914 | 46    | 0.924 |
| AlCl$_3$ | 39    | 0.961 | 51    | 0.956 | 14    | 0.973 | 45    | 0.959 | 19    | 0.968 |

Figure 7. Reswelling capability of MOG/PVA hydrogels during consecutive swelling/drying cycles.
CaCl₂ + 1 g MgSO₄ + 8 g NaCl + 20 g urea in 1000 mL deionized water and allowed to achieve swelling equilibrium at room temperature.

At certain time intervals, swollen hydrogels were withdrawn from the swelling medium, and later, excess surface water (or salt solution) was removed by filter paper and weighed accurately (Wₜ). This process was repeated until the weight of the swollen hydrogel sample remained constant (Wₑ), which demonstrated that the hydrogel had reached its equilibrium swelling capacity. The water absorbency of hydrogel (SR; g/g) was calculated by using eq 1, where (W_d) is the weight of the dry hydrogel samples. The equilibrium swelling capacity (Wₑ, g/g) was calculated using eq 2, where (W_d) is the same as eq 1.

\[
SR = \frac{W_i - W_d}{W_d} \quad (1)
\]

\[
Wₑ = \frac{W_e - W_d}{W_d} \quad (2)
\]

4.5. Water Retention Measurement. To study the percentage water retention of hydrogel samples under constant temperature, a certain quantity of water-swollen hydrogel was placed in a vacuum oven at high temperature (60 °C) and weighed every 1 h. The percentage water retention (Wₐ) was detected by the following eq 3

\[
Wₐ = \frac{W_i - W_d}{W_e - W_d} \times 100\% \quad (3)
\]

where Wᵢ is the weight of the swollen hydrogel and Wₐ and Wₑ are the same as eq 2.

4.6. Reswelling of Cross-Linked Hydrogels. A pre-weighted dry hydrogel sample was immersed in deionized water to ensure that the swelling equilibrium was achieved and weighted. The swollen hydrogels were bone-dried in a vacuum oven at 80 °C. The dried hydrogel sample was soaked again in deionized water to determine its equilibrium swelling capacity. This swelling/drying cycle was repeated five times using the same hydrogel, and finally losses of equilibrium swelling capacity were measured for each hydrogel sample.

4.7. Salt-Sensitivity Factor (f). The effect of ions (anions and cations) on the swelling capacity of the hydrogels was evaluated in various salt solutions as described by Rodrigues et al. The dimensionless salt-sensitivity factor (f) was determined using eq 4:

\[
f = 1 - \left(\frac{Wₑ}{Wₑ} \right) \quad (4)
\]

where Wₑ and Wₑ are the equilibrium swelling capacity in saline solution and in deionized water, respectively. All salts were used at concentration of 0.15 mol/L.

4.8. Network Parameters. Some important network parameters used to characterize the network structure of hydrogel were determined. The hydrogel cross-linking density (CD) is obtained by the average molar mass (Mₛ) entrapped between two neighboring cross-linking points of the polymer chain contained in the gel. The CD value is inversely proportional to Mₛ, as the higher the Mₛ, the lower the CD. According to the Flory–Huggins theory and a previously developed method, the Mₛ value of any hydrogel can be determined using eq 5:

| code | MOG % (w/v) | PVA % (w/v) | borax (mol/L) | physical condition of gel | Mₛ (g/mol) | mean pore size (μm) | SR (g/g) |
|------|-------------|-------------|---------------|---------------------------|------------|---------------------|--------|
| gel 1| 4           | 10          | 0.081 × 10⁻³  | weak                      | 3.98 × 10⁶ | 513                 | 1011.6 ± 0.7 |
| gel 2| 4           | 10          | 1.05 × 10⁻³   | moderate                  | 3.76 × 10⁶ | 497                 | 1163.5 ± 1.1 |
| gel 3| 4           | 10          | 1.31 × 10⁻³   | strong                    | 1.43 × 10⁶ | 305                 | 526.9 ± 0.4  |
| gel 4| 4           | 8           | 1.05 × 10⁻³   | moderate                  | 3.14 × 10⁶ | 453                 | 1124.1 ± 0.9  |
| gel 5| 4           | 12          | 1.05 × 10⁻³   | moderate                  | 2.02 × 10⁶ | 387                 | 601.3 ± 0.6  |

Figure 8. (a) Schematic representation for the synthesis of MOG/PVA SHs, (b) MOG powder, (c) dried hydrogel, and (d) swollen hydrogel.
\[ M_c = Q^{5/3}(D_2 V_s/(0.5 - X_1)) \]  

(5)

where \( Q \) represents the equilibrium water absorption of the hydrogel, \( D_2 \) denotes the hydrogel density, \( V_s \) is the solvent volume used for swelling, and \( X_1 \) is the Flory–Huggins interaction parameter between the solvent and hydrogel. The mean pore size or mesh size of the polymeric network of the hydrogels (\( \xi \)) broadly defines the linear distance between the macromolecular chains in a cross-linked network and is estimated using eq. 6.\(^{16–38}\)

\[ \xi = 0.071(M_c)^{1/2}/(\phi)^{1/3} \]  

(6)

Here, (\( \phi \)) is the polymer volume fraction in the swollen state.

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**Notes**

The authors declare no competing financial interest.

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