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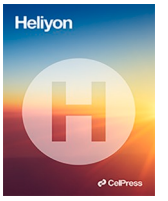

Research article

Hydroentangled waste cotton non-woven based alginate hydrogel wound dressing for high wound exudates

Faheem Ahmad [a], Anum Nosheen [a], Farooq Azam [a], Bushra Mushtaq [a], Sheraz Ahmad [a,*], Abher Rasheed [a], Yasir Nawab [a], Muhammad Sohail Zafar [b,c,d,e], Muhammad Amber Fareed [b,e,**], Moyad Shahwan [e,f]

[a] School of Engineering and Technology, National Textile University, Faisalabad, Pakistan
[b] Department of Clinical Sciences, College of Dentistry, Ajman University, Ajman, United Arab Emirates
[c] School of Dentistry, University of Jordan, Amman, Jordan
[d] Department of Dental Materials, Islamic International Dental College, Riphah International University, Islamabad, Pakistan
[e] Centre of Medical and Bio-allied Health Sciences Research, Ajman University, Ajman, United Arab Emirates
[f] College of Pharmacy and Health Sciences, Ajman University, Ajman, 346, United Arab Emirates

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ABSTRACT

Hydrogels are used in modern wound dressings due to their ability to provide comfort with quick healing. However, poor mechanical properties of hydrogels limit their availability in commercial wound dressings. Nonwovens are highly porous, strong, and flexible structures that can provide support to hydrogels without compromising their properties. In this study, a cost-effective and sustainable hydroentangled nonwoven from industrial cotton waste was prepared and incorporated into alginate hydrogel for wound dressings. The novel composite of hydroentangled cotton nonwoven and alginate hydrogel was synthesized by a simple sol-gel technique. The effect of concentration of alginate hydrogel (0.5 wt%, 1 wt%, 1.5 wt %) and drying temperature (20 °C, 40 °C, 60 °C) of composite was analyzed for high wound exudates. The properties of prepared composite samples were characterized by scanning electron microscopy (SEM), XRD, tensile strength, tear strength, Air permeability, moisture management wound exudate test, and quantitative antimicrobial testing. Moreover, the comfort performance of these samples was evaluated by air permeability and moisture management testing. The properties of developed composites are highly dependent on the concentration of alginate and drying temperature. The results showed that maximum fluid absorbency (%) of 650 was achieved with good comfort properties. This study can help to increase the commercial availability of hydrogel-based wound dressings.

1. Introduction

Hydrogels are 3D polymeric networks capable of retaining huge amounts of fluids. These materials are of significant interest due to their high water content, tunable properties, and biocompatibility which make them ideal for a range of applications, particularly in biomedicals [1–3]. Several studies have revealed that hydrogels can mimic natural tissues, providing a moisture environment that

* Corresponding author.
** Corresponding author. Department of Clinical Sciences, College of Dentistry, Ajman University, Ajman, United Arab Emirates.
E-mail addresses: sheraz@ntu.edu.pk (S. Ahmad), m.fareed@ajman.ac.ae (M.A. Fareed).

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promotes cell migration and proliferation, making them especially effective in wound healing and other medical applications [4–7]. The hydrogels are used for drug delivery systems, and tissue engineering, as scaffolds for cell growth and wound dressings [8–10]. The criterion for modern wound dressing is to protect the wound and accelerate the healing process. Hydrogels fulfill this criterion by providing a moist healing environment, absorbing exudate, and cooling the wound [11–13]. Additionally, their ability to load therapeutic and antimicrobial agents enhances the healing process. Natural polymer-based hydrogels such as chitosan, alginate, cellulose, and gelatin are known for their biocompatibility whereas synthetic polymeric hydrogels like polyvinyl alcohol (PVA), polyacrylamide (PAAm) and polyethylene glycol (PEG) are used due to better mechanical properties [14,15]. The composites of natural and synthetic hydrogels offer excellent properties with the freedom to incorporate various kinds of nanoparticles and drugs suitable for wound healing. These composites are produced in the form of gels, fibers, sheets, and nonwoven fabrics [7,16]. Nonwovens are versatile fabrics made directly from fibers as compared to conventional fabrics. These fabrics are commercially used in several applications such as insulation, filtration, and biomedicals due to ease of production and cost-effectiveness. The flexibility, breathability, and high absorbency make them highly suitable for face masks, surgical gowns, and wound dressings [17–20]. In the recent past, various studies concluded that a combination of nonwovens and hydrogels is highly efficient for wound dressing, especially for high wound exudates. Cotton, wool, hemp nonwovens, and alginate, cellulose-based hydrogels are used for wound dressings. The properties of these composites depend on the type of fiber, hydrogel, the structure of nonwoven, concentration of hydrogel, and drying temperature [21]. Usually, these composites are prepared by sol-gel methods, in which the needle-punched nonwovens are used as a substrate and soaked into a solution of hydrogel followed by drying at room temperature [22–25]. The needle-punched nonwovens are durable, thick, and strong fabric suitable for wound dressing. However, the needle-punched nonwovens are rigid and less comfortable to body contours which might reduce the patient comfort [26,27]. Therefore, there is a need to use other nonwovens such as hydroentangled which is more flexible, highly absorbent, and comfortable [28,29]. In this study, a hydroentangle nonwoven was prepared from textile industrial waste and incorporated with alginate hydrogel to form a unique sustainable system for wound dressings. Alginate is a natural polymer which is suitable for wound dressing due to its biocompatibility and good moisture retention ability which helps to promote the healing process. In this work, the quantification of antimicrobial properties, the effect of alginate hydrogel concentration, and drying temperature were analyzed to optimize the properties of composite for wound dressings. The results of this study may help to prepare commercial hydrogel-based nonwoven wound dressing for high wound exudates.

2. Materials and methods

2.1. Materials

Sodium alginate, $C_6H_7NaO_6$ (>97 % pure), Calcium chloride, and $CaCl_2$ (>95 % pure) were procured from Daejung Chemicals, Korea. The non-woven cotton fabric of GSM (–) was kindly provided by the National Textile University, Faisalabad, Pakistan.

2.2. Experimental

2.2.1. Preparation of cotton non-woven by hydroentanglement process

In this study, a hydroentangled non-woven was prepared by using industrial cotton waste fibers of 18–20 mm in length. Firstly, the pre-wet cotton fibers were passed through the Trutzschler's fiber opening line to provide a regular feed to the card machine. A double-doffer carding machine was used to prepare a parallel-laid cotton web. This prepared cotton web was bonded by the Fliessner hydroentanglement process at AquaJet which has six water injectors to provide pressure. A jet pressure of 9 MPa was given to form a uniform and fine non-woven structure of cotton. A uniform web speed of 25–30 m min^{-1} was fixed throughout the fiber entanglement process. The areal density of cotton non-woven was fixed at 55 g/m^2.

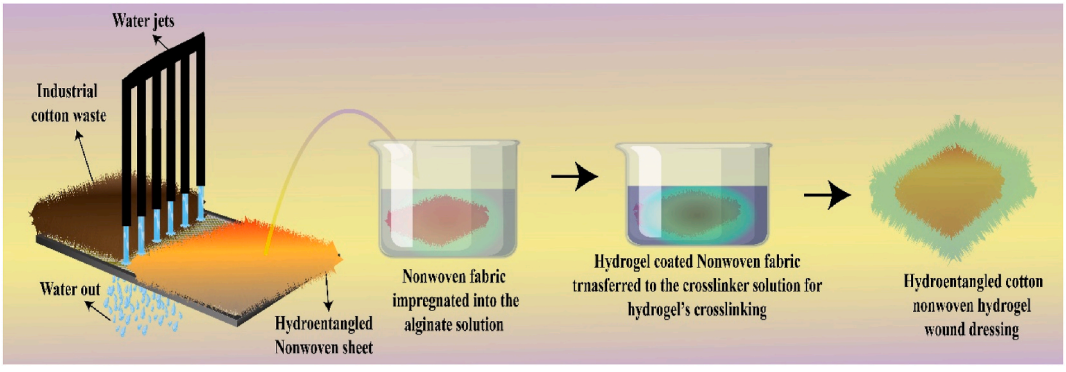

Fig. 1. Schematic of preparation of cotton non-woven/alginate hydrogel composite.

2.2.2. Preparation of hydrogel composites

Three aqueous solutions of sodium alginate of different concentrations 0.5 %, 1 %, and 1.5 % were prepared by adding the selected concentrations of sodium alginate in 500 mL of distilled water. The as-prepared solutions were subjected to magnetic stirring at 900 rpm and the stirring was continued overnight at room temperature. After that, the as-prepared solutions were applied to the non-woven fabric as shown in Fig. 1. For this purpose, three square-shaped non-woven samples of dimensions 12 × 12 inches were placed on separate petri dishes followed by the addition of all three solutions in respective plates. The fabric samples were completely immersed in solutions and kept at room temperature for 30 min. Meanwhile, 0.2M calcium chloride ($CaCl_2$) solution was prepared, and the resultant solution was stirred at 900 rpm and the stirring was continued for 30 min at room temperature. After that, the fabric samples were removed from sodium alginate solutions and immersed in $CaCl_2$ solution for 30 min. After gel formation, the non-woven fabric samples impregnated with sodium alginate were dried for 24 h in an oven at three different temperatures i.e., 20, 40, and 60 °C.

The full factorial design was used to investigate the effect of concentrations of sodium alginate and drying temperatures on each response variable. The design of the experiment for the prepared samples is shown in Table 1.

2.3. Characterization

The morphological properties of developed non-woven samples were characterized through a FEI Nova Nano scanning electron microscope (SEM). The prepared non-woven samples were coated with gold before visualizing them through SEM images. The crystalline structure of the developed samples was analyzed through X-ray diffraction. X-ray diffraction (XRD) spectra were recorded from 10° to 80° with a diffractometer (Bruker, German). The scanning rate was 1°/min and 40 KV accelerating voltage was applied. ISO 20743 transfer method standard testing protocol was followed for quantitative antibacterial analysis.

2.3.1. Mechanical properties assessment

The equipment LLOYD universal tensile strength tester was used to characterize the tensile strength/elongation% of the samples according to the ASTM D5035-19 Grab method. Each specimen was cut to a 10 mm length and 5 mm width dimensions. A 100 N load cell operating at a constant rate of 0.1 mm/s was used to conduct tensile strength testing at a speed of 10 mm/min and a gauge length of 10 mm. The experiment was repeated 3 times and the mean value was recorded. For tear strength measurement, the ELMENDORF tearing strength, UTS manufacturer machine was used and tested according to ASTM D 5587. At first, the sample is cut to the initial dimensions of 3″ × 4″, but an additional cut of 12 × 15 mm is placed at the center and top of the sample in both the weft and warp directions of the sample. The experiment was performed 3 times and the mean reading was recorded.

2.3.2. Physico-comfort properties assessment

The physical-comfort properties i.e., air permeability, and moisture management test were evaluated. Air permeability (AP) is a measure of the rate of airflow through a known area of fabric under prescribed pressure. It provides information about the breathability of a fabric. The air permeability (AP) of the untreated and treated cotton fabric was evaluated through the Air Permeability Tester MO21A (SDL ATLAS) by the ISO-9237 standard testing method. The clamping arm of this instrument presses the fabric sample to secure it over the testing head. This action turns on the vacuum pump and testing pressure is maintained. The test was performed at a pressure of 100 pa and 20 cm^2 area. The experiment was repeated 10 times, and the mean value was determined. The moisture management test (MMT) was measured on the SDL ATLAS Moisture Management Tester by the AATCC-195 standard testing method. For this, a sample having dimensions of 3" × 3" was taken. Five readings were recorded, and the average value was calculated. EN 12,726–1:2002 standard test method was used for the characterization of wound exudate absorbing properties. An aqueous solution containing 0.368 g of ($CaCl_2$ x $2H_2O$) Calcium chloride dehydrate, 2.298 g of Sodium chloride (NaCl), and 1 L of water was pre-heated at 37 °C. All the developed samples were cut in dimensions of 3 × 3 cm, weighed, and put in the petri dishes as presented in Fig. 3. Then the solution on the sample corresponding to 100 times its mass and placed in a laboratory oven at 37 °C. After 2 h, the samples were removed from the oven and hung with the tweezer for 30 s for excessive fluid removal. Then the wet samples and fluid absorption were calculated as given below:

$$\text{Fluid handling capacity\%} = \frac{(\text{wet weight of sample} - \text{dry weight of sample})\ \text{X}\ 100}{\text{Dry weight of sample}}$$

Table 1
Design of experiment (DOE) for the development of hydrogel composites.

Sr. No	Sample code	Concentration of sodium alginate (%)	Drying temperature (°C)	Drying time (hours)	Thickness (mm)
1.	S1	0.5	20	24	0.68 ± 2.11
2.	S2	0.5	40	24	0.68 ± 1.21
3.	S3	0.5	60	24	0.62 ± 2.45
4.	S4	1	20	24	0.69 ± 1.98
5.	S5	1	40	24	0.71 ± 2.67
6.	S6	1	60	24	0.69 ± 1.88
7.	S7	1.5	20	24	0.75 ± 2.15
8.	S8	1.5	40	24	0.78 ± 1.98
9.	S9	1.5	60	24	0.78 ± 1.67

2.3.3. Quantitative antibacterial analysis

Quantitative antibacterial analysis was performed for four samples i.e., control, S7, S8, and S9. For this, 2 swatches of each sample were taken and placed on the agar plates previously inoculated with 1 ml of gram-positive bacterial strain (*S. aureus*). One of the specimens was neutralized and diluted in broth solution followed by plating the solution on agar plates to obtain bacterial count at 0 h. The remaining swatch of each sample was incubated in the oven at standard incubation conditions (37 °C for 24 h) overnight. The next day, the same plating procedure was performed to get the bacterial count after 24 h. The following equations were applied for the calculation of antibacterial activity in terms of log reduction and percentage reduction.

$$\text{logreduction} = \text{F-G} \tag{1}$$

Here, F= (log C_t - logC_0) and G= (log T_t – log T_0), C_0 and C_t are the bacterial counts of control 0h and after 24h respectively while T_0 and T_t are the bacterial counts of the treated sample at 0h and after 24 h. Percentage reduction was calculated according to Equation (2).

$$\%\text{Age Reduction} = [(\text{logCt-logTt}) / \text{logCt}]^* 100 \tag{2}$$

3. Results and discussion

3.1. Scanning electron microscopy

Scanning electron microscopy (SEM) analysis was performed to assess the morphological characteristics of developed hydrogel composites to study the effect of an increase in the concentration of sodium alginate hydrogel and increasing drying temperature. To study the effect of an increase in the concentration of sodium alginate, the SEM images of samples S1 (0.5 % concentration), S4 (1 % concentration), and S7 (1.5 % concentration) were recorded, and the images are provided in Fig. 2a. The increase in concentration of sodium alginate has sufficiently altered the morphology and surface characteristics of hydrogel composites. The SEM image of samples S1 and S4 (lower concentration) has a less compact structure. This is because the lower concentration of sodium alginate leads to a less dense network with few sites in polymer sites available for cross-linking. As a result, the hydrogel composites might have more open spaces, resulting in a less compact structure and higher porosity. Whereas, the SEM images of sample S7 having the highest concentration of sodium alginate have shown smooth and fine surface morphology. It is attributable to the higher concentration of sodium alginate which offers more polymer chains available for cross-linking with Ca^{2+} ions from calcium chloride. This enhanced polymer density leads to a highly interconnected network, which reduces the porosity and makes the surface of hydrogel composites smoother.

On the other hand, the SEM images of samples S4 (dried at 20^0 C), S5 (dried at 40^0C), and S6 (dried at 60^0 C) were recorded to observe the effect of varying drying temperatures on the morphological and structural characteristics of hydrogel composites (Fig. 2b). The increase in drying temperature also has influenced the surface characteristics of hydrogel composites. It is seen that sample S4 dried at 20^0 C with a greater number of pores while sample S5 dried at an intermediate temperature (40^0 C) has shown very few pores and has compact areas on its surface. Whereas sample S6 which dried at a higher temperature (60 °C) has shown a combination of rough, compact, and porous structures. Drying of hydrogel composites at lower temperature slows down the water evaporation. The slowed drying process leads to the generation of larger pores within the hydrogel structure resulting in higher porosity. The drying of hydrogels at an intermediate temperature balances the water evaporation rate leading to a moderate level of porosity. This could yield a structure with a combination of porous as well as compact regions. Whereas drying hydrogels at higher temperatures speed up the rate of water evaporation. This accelerated drying can trap water within the hydrogel leading to the formation of smaller pores or even defects and cracks due to the rapid water escape resulting in a more variable morphology, including a combination of rough and compact areas [30,31].

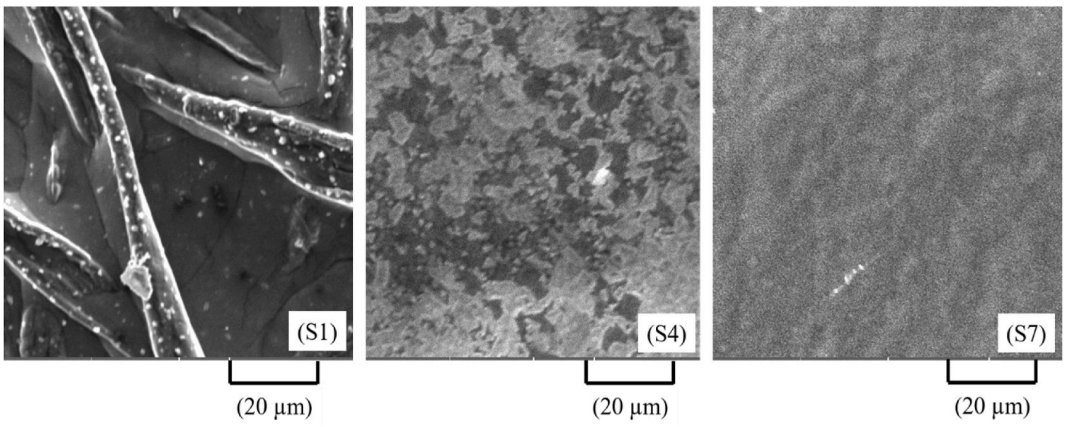

Fig. 2a. SEM images of samples S1 (0.5 %), S4 (1 %), and S7 (1.5 %).

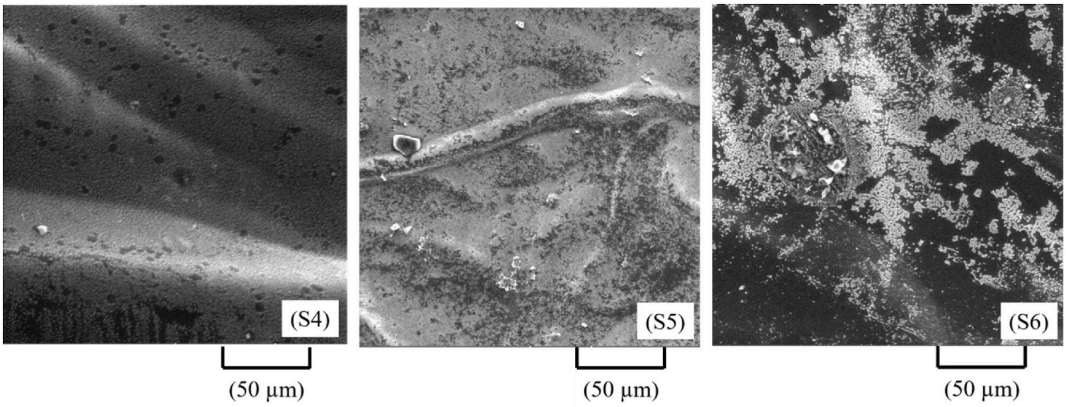

Fig. 2b. SEM images of samples S4 (20^0C), S5 (40^0C), and S6 (60^0C).

3.2. X-ray diffraction analysis

X-ray diffraction was used to determine the crystalline and amorphous region of the sodium alginate hydrogel composites. As all the developed samples are composed of sodium alginate which is a biopolymer, and it has an amorphous structure. Amorphous materials lack a repeating crystalline lattice structure due to which sharp XRD peaks are produced. Instead, the polymer chains are randomly arranged, resulting in a diffuse or broad scattering pattern rather than well-defined peaks. Therefore, the XRD spectra of all the samples are not recorded. The XRD spectra of samples S7 and S9 were recorded as these samples have shown the optimum results. The XRD of samples S7 and S9 was performed, and the results are depicted in Fig. 3. From the XRD pattern, it was observed that there was only one crystalline region peak at 22° in the XRD spectra of S7 (left image) while no sharp peak was observed in the XRD spectra of samples S9 in this region. Moreover, the remaining regions are amorphous as no other sharp signal was observed in the spectrum. These observations suggested that the amorphous region was increased with the increase in the temperature from 20 °C to 60 °C for samples S7 and S9. The reason that no sharp peaks were observed at higher drying temperatures (60 °C) might be due to increased amorphousness induced by the elevated temperatures. The drying temperature has a significant influence on the structure of the resulting material. Higher drying temperatures can lead to more amorphous structures due to which the XRD spectra of sample 9 have shown more scattered signals. Conclusively, the XRD spectra revealed that the major portion of the sodium alginate hydrogel is amorphous due to which no sharp peaks were observed. Although sodium alginate has some degree of crystallinity, it is not sufficient to produce sharp and strong XRD peaks. The absence of sharp peaks in the XRD spectra of sodium alginate and calcium chloride hydrogel composites could be due to a combination of factors, including the amorphous nature of sodium alginate, low crystallinity, and varying drying temperatures.

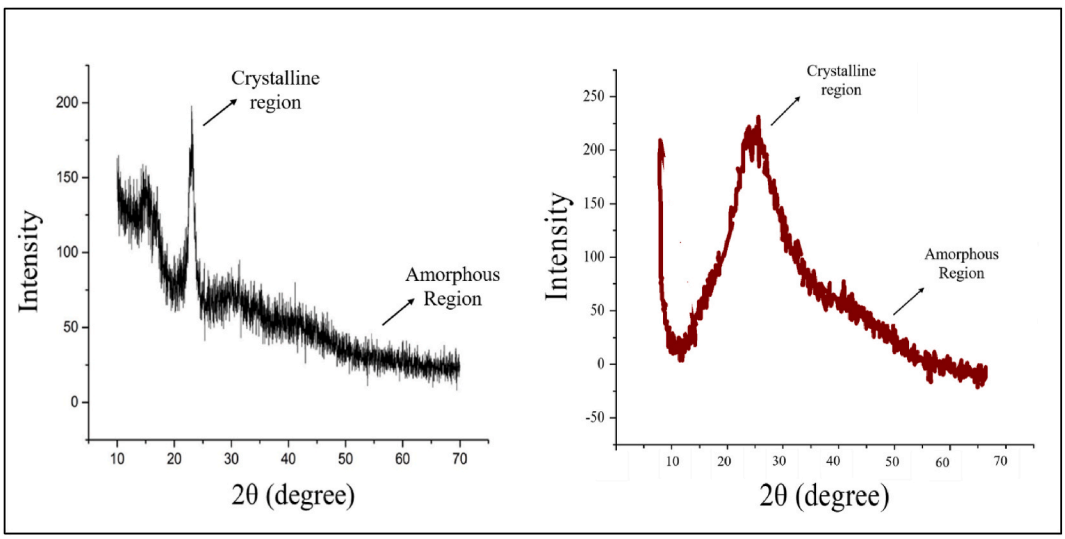

Fig. 3. XRD spectra of sample S7 and S9.

3.3. Air permeability

The air permeability (AP) of a nonwoven sample is affected by a variety of parameters, including the concentration of hydrogels, drying temperature, type of raw material used, the method used to fabricate the fabric, and any chemical finishes that may be present [32,33]. The air permeability values for the developed hydrogel composites were determined and the obtained results are presented in Fig. 4. From the figure, it was observed that the air permeability values (mm/sec) were decreased with the increase in the concentration of sodium alginate i.e., minimum of 0.5 % concentration and maximum values of 1.5 % concentration. As the concentration of sodium alginate was increased (from 0.5 % to 1 %–1.5 %), it to the formation of a denser hydrogel structure. This is because higher concentrations of sodium alginate result in more polymer chains as well as cross-linking leading to the reduced spaces between them. The reduced space between the polymer chains and cross-linking reduces the porosity of the hydrogel which in turn decreases the porosity. The decreased porosity restricts the flow of air through the hydrogel composites, leading to decreased air permeability. Furthermore, the results have also suggested that varying the drying temperature has also significant effect on the air permeability of hydrogel composites. The air permeability was observed to decrease with the increase in the temperature, For instance, the air permeability of sample S7 was 17 mm/s which was decreased to 8 for sample S9 when the temperature was increased from 20 °C to 60 °C. These findings could be best explained based on the fact that the drying temperature could have a significant effect on the rate of cross-linking and gelation of the sodium alginate. Higher temperatures accelerate these processes, resulting in a denser network of cross-linked polymer chains in hydrogel composites [34,35]. The microstructure of hydrogel is critical in determining its air permeability and higher drying temperatures could lead to the formation of a less porous and more compact structure, reducing the air pathways and restricting the air flow through them. The combined effect of sodium alginate concentration and drying temperature caused a pronounced decrease in the air permeability of hydrogel composites. Higher concentrations have already reduced the porosity, whereas the higher drying temperatures made the structure even denser resulting in the reduction in air permeability.

3.4. Tensile strength

The tensile strength of the developed hydrogel composites was determined according to the standard testing protocols to study the effect of the sodium alginate concentration and drying temperatures. The tensile strength of all the samples is shown in Fig. 5. It is obvious from the figure that the tensile strength of the hydrogel composites has increased with the increase in the concentration of the sodium alginate i.e., 57 N, 71 N, 83 N for samples S1, S5, and S7, respectively. Whereas the temperature has shown the inverse relation with the tensile strength of the samples. The tensile strength of hydrogel composites has decreased with the increase in temperature i.e., 40 N, 54 N, and 61 N for samples S3, S6, and S9, respectively. Our findings where the tensile strength of the hydrogel composites composed of sodium alginate and calcium chloride was highest with the higher concentration of sodium alginate and at lower drying temperatures, are perfectly aligned with the basic principles of hydrogel formation as well as polymer chemistry. The higher concentration of sodium alginate results in a dense network of polymer chains in the hydrogels which make the hydrogels stiffer and less flexible, which can lead to higher tensile strength. Whereas the lower concentrations of sodium alginate led to a less dense network of polymer chains resulting in a more flexible and elastic hydrogel. The enhanced elasticity could result in lower tensile strength as the hydrogel is more prone to breaking as well as deformation under tension. Furthermore, lower drying temperature during the hydrogel formation allows the slow cross-linking between calcium chloride ions and sodium alginate resulting in a more ordered and uniform structure in the hydrogel, which is attributed to the enhanced tensile strength at lower temperatures [36,37] Whereas the increasing drying temperature accelerates the cross-linking process during the hydrogel formation. The accelerated cross-linking might cause a less uniform structure within the hydrogel. As a result, the hydrogel composites might become less resistant to stretching and have lower tensile strength [38,39].

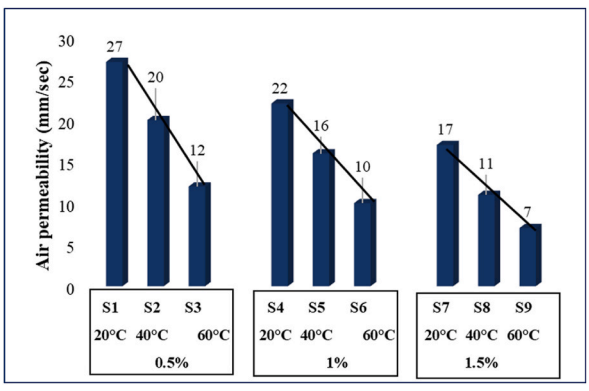

Fig. 4. Air permeability values of samples S1-S9.

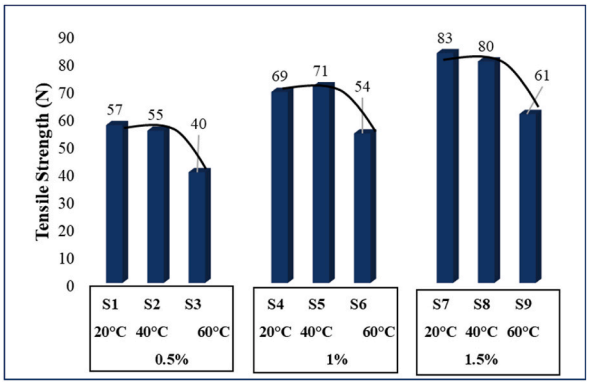

Fig. 5. Tensile strength values of Samples S1-S9.

3.5. Tear strength

The tear strength of the developed hydrogel composites was determined according to the standard testing protocols to study the effect of the sodium alginate concentration and drying temperatures. The tear strength of all the samples is shown in Fig. 6. It can be seen from the graph that the concentration of sodium alginate and drying temperature has a direct relation with the tear strength. The tear strength has increased with the increase in the concentration of hydrogel as well as an increase in the temperature. The tear strength of sample S1 (0.5 % sodium alginate) was 21 N which was increased to 41 N for sample S7 (1.5 % sodium alginate). Similarly, the tear strength for sample S7 was 41N when the drying temperature was 20 °C, which increased to 53 for sample S9, which was dried at 60 °C. The current findings could be explained by increasing the sodium alginate concentration results in a stronger hydrogel. Therefore, the tear strength significantly increased as the concentration was increased from 0.5 % to 1 %–1.5 % sodium alginate. Furthermore, higher drying temperatures could lead to denser hydrogel structure due to enhanced cross-linking resulting in increased tear strength as the temperature is increased from 20 °C to 40 °C–60 °C.

3.6. Wound exudates

Wound exudate absorption is the primary factor considered when designing wound dressings to provide an adequately moist environment [40]. The fluid absorbency (%) was calculated for all the developed hydrogel composites samples and the wound exudate absorbing characteristics vs. time (monitored for 24 h and readings were taken at 6 different intervals i.e., 4h, 8h, 12h, 16h, 20h, and 24h) have given in Fig. 7. The results indicated that the fluid absorbency was significantly increased with the increase in the concentration of sodium alginate hydrogels and drying temperature. This suggests that wound exudate properties of hydrogel composites are significantly influenced by the concentration of sodium alginate as well as drying temperature. Higher concentrations of sodium alginate result in hydrogels with enhanced absorption capacity. This is because the higher concentration of alginate offers more absorbing sites in polymer to absorb and retain wound exudate. Also, the increase in the concentration of sodium alginate typically leads to hydrogels with enhanced mechanical strength which is crucial for maintaining the structural integrity of the hydrogel when it absorbs wound exudate. Moreover, drying at higher temperatures leads to increased porosity in the hydrogel structure which enhances the absorption capacity by generating more pathways for fluid to enter the hydrogel. High temperatures drying could also potentially result in a more cross-linked and mechanically robust hydrogel structure which also has a positive effect on the wound exudate

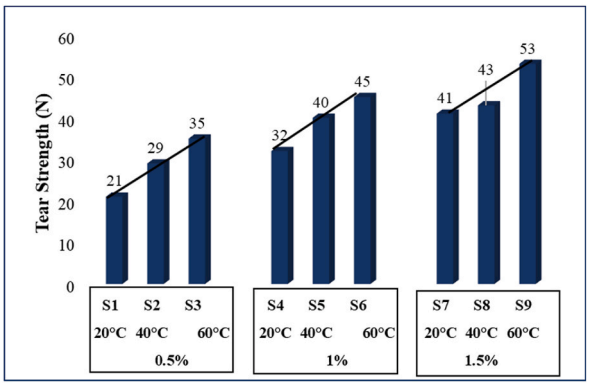

Fig. 6. Tear strength values of samples S1-S9.

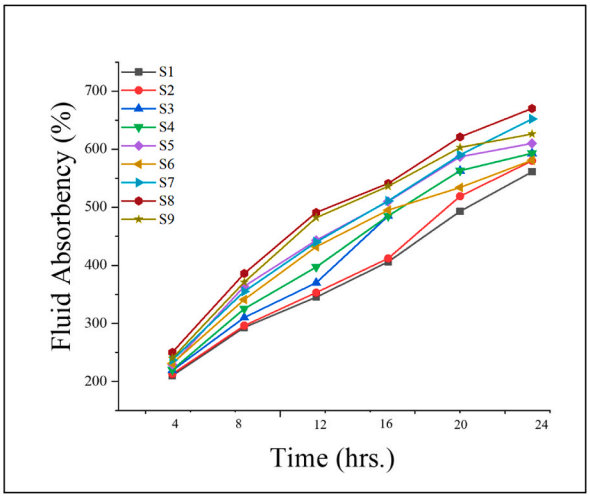

Fig. 7. Wound exudates analysis results at different time intervals.

properties of the hydrogel composites at higher temperatures. Thus, the results showed that the developed hydrogel had a strong capability for absorbing and retaining water, and it also had an adequate capacity for wound exudate absorption.

3.7. Moisture management properties

The overall moisture management capability (OMMC) of the hydrogel composites was evaluated for all the hydrogel composites samples to analyze the effect of sodium alginate concertation of drying temperature on the OMMC of samples. The obtained results are presented in Fig. 8. The graph clearly shows that the OMMC of the hydrogel composites was increased with the increase in the concentration of sodium alginate hydrogels i.e., 0.55 for S1 (0.5 % concentration) and 0.71 for samples S7 (1.5 % concentration). This suggested that the concentration of sodium alginate has a direct relation with the absorption capacity of the hydrogel composites. As the concentration of sodium alginate increased, the capacity of the hydrogel composites to absorb moisture from the surrounding environment also increased. It is attributed to the higher sodium alginate concentration which has created a dense network structure within the hydrogels which in turn can trap more water molecules resulting in enhanced absorbency. Increased concentration of sodium alginate concentrations has also increased the swelling behavior of the hydrogel due to which it retains more water. Moreover, it was observed that the relation between increasing drying temperature and moisture management properties of the hydrogel composites is not linear. The OMMC values were increased first with the increase in the temperature i.e., 0.55 for sample S1 (20 °C) was increased to 0.67 for sample S2 (40 °C). This could be explained on the basis that the drying temperature influences the pore size and porosity of the hydrogel composites. Higher drying temperatures lead to highly interconnected and larger pores which can enhance the moisture management properties of the hydrogel composites. However, a sharp decline in the OMMC values was observed with the further rise in temperature from 40 °C to 60 °C. The OMMC value of sample S2 was 0.67 which was dropped to 0.16 for sample S3 which was dried at 60 °C. This sudden and unexpected decrease in OMMC values at higher temperatures could be due to the reason that when the drying temperature increases, the evaporation rate of water from the hydrogel is also increased. The increased water

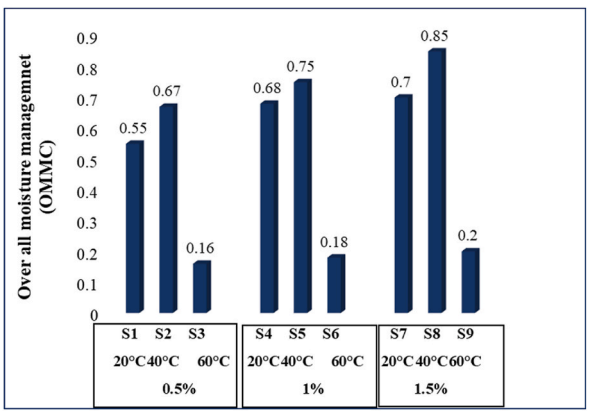

Fig. 8. Moisture management properties of samples S1-S9.

evaporation rate can result in faster drying leading to non-uniform drying as well as the formation of cracks in the hydrogel structure. Excessive drying temperatures damage the structure of hydrogel composites. As a result, the hydrogel composites may become brittle and more prone to defects or cracking compromising the moisture management liquid management properties of hydrogel composites (see Fig. 9).

3.8. Statistical analysis

The statistical analysis of the obtained results was performed through MINITAB 18 statistical software and the effect of concentration of sodium alginate and drying temperature was assessed. The p-values for all responses against each factor are presented in Table 2. With 95 % confidence, responses which have p-values less than 0.05 are statistically significant. In case of both variable factors i.e., concentration of sodium alginate concentration and drying temperature, all responses which have a p-value less than 0.05 suggest a significant effect whereas the other responses which have p-values greater than 0.05 have an insignificant effect. The regression equations for each response variable, including terms that are statistically significant, are provided in Table 2. The R2 values demonstrate the percentage of response variable variation that can be explained by the terms or factors in the regression equations. In Table 2, statistically significant responses are represented by an asterisk (*) symbol, while a minus (−) sign indicates that the response value increases as the factor value drops and vice versa, whereas higher coefficient values indicate a stronger impact on the corresponding term.

3.9. Quantitative antibacterial analysis

ISO 20743-transfer method was performed for the quantitative antibacterial analysis. In our previous research, we have documented that sodium alginate hydrogel composites have antibacterial properties. We have established through qualitative antibacterial analysis that significant antibacterial activity could be achieved with 1.5 % concentration of sodium alginate. The antibacterial action was not significant below these concentrations. In the current study, the hydrogel composites samples were tested through quantitative antibacterial analysis for more accurate assessments. The sample S7-S9 was tested as significant antibacterial activity was achieved with 1.5 % concentration of sodium alginate (previous research). Fig. 9a and b shows the antibacterial activities of samples S7-S9 in terms of percentage reduction and log reduction, respectively.

All three samples have shown excellent antibacterial activity and inhibited more than 80 % of inoculated bacterial colonies. The log value was also remarkably reduced from 6.41 (control sample) to 0.69 and 0.47 for samples S7-S9 which suggests >80 % reduction in bacterial concentration.

For more clarification, the images of agar plates showing the growth of bacterial colonies are given in Fig. 10. It is evident from the image there are a great number of bacterial colonies in agar plate of control samples whereas the bacterial colonies are remarkably reduced for the hydrogel composite samples S7 (1.5 % concentration). Thus, the obtained results have revealed that the developed hydrogel composites have excellent antibacterial activity and the antibacterial activity of all three samples was comparable. Hence, it was concluded that the drying temperature does not have any significant impact on the antibacterial activity of the hydrogel composites. The strong antibacterial action could be attributed to the intrinsic antibacterial action of calcium chloride with sodium alginate. When calcium chloride reacts with sodium alginate, calcium alginate is formed which is believed to have antibacterial action.

4. Conclusions

The alginate hydrogel incorporated hydroentangled cotton non-woven was successfully prepared by using sol-gel method for wound dressings. The prepared samples of hydrogel composite were highly flexible with good mechanical strength. The properties of these developed hydrogel composites were significantly affected by the alginate concentration and drying temperature. It was observed that the air permeability of composites was decreased with the increase in the concentration of sodium alginate i.e., minimum of 0.5 % concentration and maximum values for 1.5 % concentration. Moreover, the air permeability was observed to decrease with the increase in the temperature. The air permeability of 17 mm/s was decreased to 8 mm/s for inclination of drying temperature from 20 °C to 60 °C. The relationship of increasing drying temperature and moisture management property (overall moisture management capacity, OMMC) of the hydrogel composites is not linear. However, the OMMC of the alginate hydrogel composites was increased with the increase in the concentration of sodium alginate. The wound exudate absorption capacity of developed composites was increased by increasing both alginate concentration and drying temperature. The maximum fluid absorbency (%) of 650 was achieved without damaging the comfort properties of hydrogel composite for wound dressings. The developed hydrogel composites have excellent antibacterial activity and the antibacterial activity at alginate concentration of 1.5 % with drying temperature from 20 °C to 60 °C. It was concluded that the prepared alginate hydrogel composite by using wasted industrial cotton has good properties to be potentially used in commercial wound dressing applications.

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Table 2
p-values of input variables and regression equations (R^2) for output variables.

p-values		
Properties of hydrogel composites	Concentration of sodium alginate	Drying temperature
Air permeability	0.000*	0.000*
Tensile strength	0.000*	0.000*
Tear strength	0.020	0.000*
OMMC	0.414	0.031
Regression equation and R^2 values		
Properties of hydrogel composites	**Regression equation**	**R^2**
Air permeability	59.33 + 23.00 Concentration of sodium alginate - 0.5000 Drying temperature (°C)	97.18
Tensile strength	6.33 + 16.33 Concentration of sodium alginate + 0.3750 Drying temperature (°C)	98.09
Tear strength	34.17–6.17 Concentration of sodium alginate - 0.2792 Drying temperature (°C)	96.42
OMMC	0.846 + 0.143 Concentration of sodium alginate - 0.01150 Drying temperature (°C)	60. 02

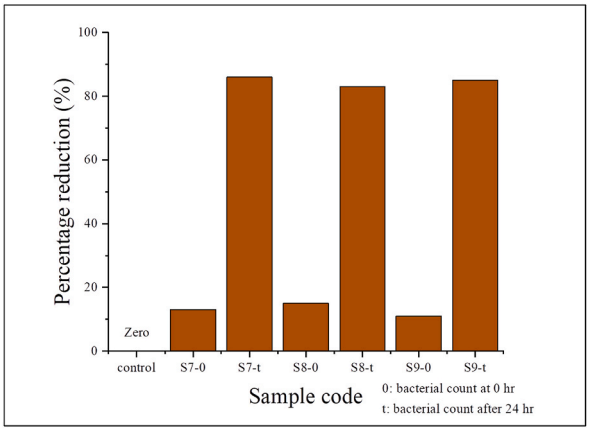

Fig. 9a. Percentage reduction values of hydrogel composites samples (S7-S9).

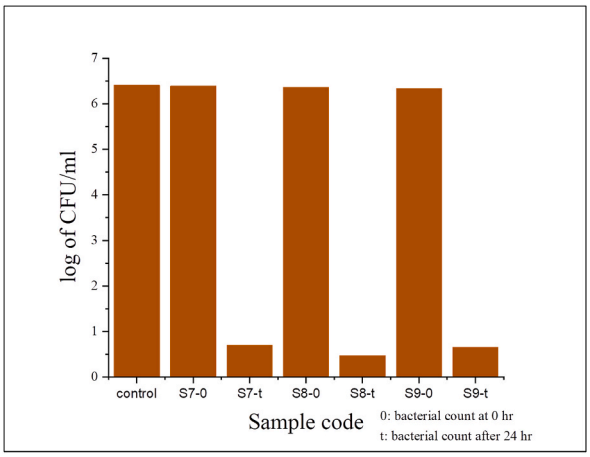

Fig. 9b. Antibacterial activity of samples S7-S9 in terms of log of (CFU, colony forming units) (CFU/ml).

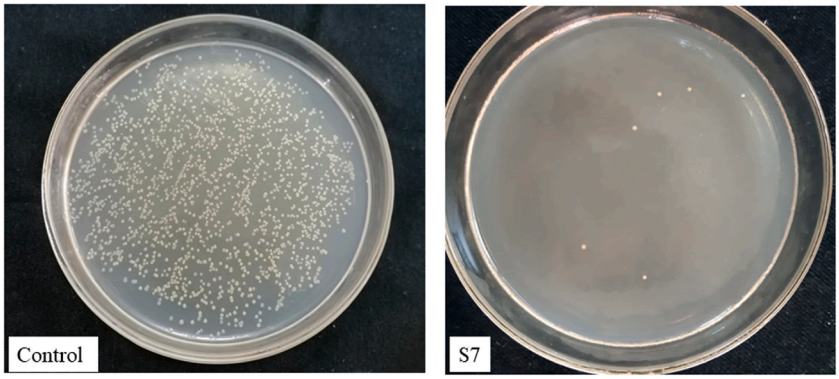

Fig. 10. Agar plates images for the control sample and hydrogel sample S7 after 24 h.

Conflicts of interest/Competing interests

The authors declare that they have no conflict of interest.

Availability of data and material

The data will be made available as per requirement.

Code availability

Not applicable.

Ethics approval

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Faheem Ahmad: Writing – review & editing, Data curation. **Anum Nosheen:** Software, Methodology, Conceptualization. **Farooq Azam:** Writing – original draft, Software, Methodology, Data curation, Conceptualization. **Bushra Mushtaq:** Writing – original draft, Data curation. **Sheraz Ahmad:** Writing – review & editing, Supervision, Funding acquisition. **Abher Rasheed:** Validation, Software. **Yasir Nawab:** Validation, Supervision, Software. **Muhammad Sohail Zafar:** Visualization, Investigation, Funding acquisition. **Muhammad Amber Fareed:** Validation, Software, Funding acquisition. **Moyad Shahwan:** Writing – review & editing, Visualization, Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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