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Development and characterisation of superabsorbent fibrous web for hygiene applications

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This study focuses on developing superabsorbent fibrous web using sodium carboxymethyl cellulose (Na CMC), polyvinyl alcohol (PVA), and citric acid (CA). The superabsorbent polymer solution is electrospun into a fibrous web with varying CA concentrations (0 %, 2 %, 4 %, 6 %, 8 %, and 10 %). Then, the morphology and chemical functional groups of the fibrous webs have been analysed using scanning electron microscopy and Fourier-transform infrared spectroscopy. The liquid absorption properties, such as free absorption capacity, liquid retention capacity, absorption under load, and structural stability of the webs, have been evaluated using distilled water, saline solution, and synthetic blood. The fibrous web containing 4 % CA (CA4: 10 % Na CMC, 10 % PVA, and 4 % CA) exhibits maximum absorption capacity among the tested samples. The absorption properties of CA4 sample have also been compared with those of a commercially available sanitary napkin. Furthermore, the crosslinked CA4 sample exhibits better structural stability compared to the non-crosslinked sample.

Keywords: Citric acid, Electrospun fibrous web, Hygiene application, Polyvinyl alcohol, Sodium salt carboxymethyl cellulose, Superabsorbent

1 Introduction

The demand for hygiene textiles has steadily increased due to their vital role in maintaining personal hygiene and providing comfort. Hygiene textiles, a subset of medical textiles, are specifically designed to absorb biofluids such as blood, urine, and menstrual fluid. In general, cellulose fibres are used in these applications due to their good liquid absorbency and soft texture. However, in products like sanitary napkins and diapers, the liquid absorption capacity of these fibres is often inadequate. Superabsorbent polymers (SAPs) have been developed to address this limitation as highly efficient liquid-absorbing materials. With three-dimensional crosslinked structure and hydrophilic functional groups, these polymers can absorb over 10 g/g of liquid, outperforming conventional absorbents such as wood pulp and cotton fibres[1,2]. Therefore, SAPs are widely used in hygiene products, such as sanitary napkins and diapers, because of their superior liquid absorption properties. However, the non-biodegradable nature of most commercial SAPs has raised environmental concerns, as their widespread disposal contributes to the accumulation of waste. This challenge has driven research into biodegradable SAPs that offer comparable performance while reducing environmental impact. In recent years, significant efforts have been made to develop biodegradable superabsorbent polymers (SAPs) using cellulose and its derivatives[3]. For instance, studies have reported the development of SAPs from materials such as lyocell and chitosan, which are suitable for absorbent products[4,5]. However, these alternatives are often cost-prohibitive, making them inaccessible for widespread use. Therefore, this study focuses on developing cost-effective and biodegradable SAP using sodium carboxymethyl cellulose (Na CMC) and citric acid (CA). Na CMC is a renewable, biocompatible, and biodegradable polymer[6]. It is known for its high liquid absorption property due to its polyelectrolyte nature and presence of anionic (COO^-) groups. Citric acid, a widely available organic acid, is used as a crosslinking agent to convert Na CMC into a water-insoluble SAP[1].

This study also uses electrospinning technique to explore the fabrication of Na CMC-based SAP in the form of a fibrous web. Electrospinning is a simple technique that produces fine fibres with a high surface area-to-volume ratio and porosity[7-9], making them suitable for absorbent applications. However, conventional single-needle electrospinning is limited by its low production rate, hindering industrial use scalability. The needleless electrospinning[10,11]

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overcomes this limitation by generating fibres through multiple jets simultaneously, significantly improving the production rate while preserving fibre quality. This advancement makes electrospun fibres more practical for large-scale manufacturing, particularly in hygiene-absorbent products[12]. However, the electrospinning of Na CMC poses challenges due to its rigid molecular structure and ionic group repulsion behaviour[13,14]. To overcome this, Na CMC was blended with polyvinyl alcohol (PVA), a biocompatible and biodegradable polymer[15,16] that enhances the spinnability of Na CMC by reducing repulsive forces among anions (COO^-). FTIR, SEM, and absorption tests, including liquid retention capacity, free absorption capacity, and absorption under load, investigated the properties of electrospun samples. Furthermore, the absorption performance of the developed fibrous webs was compared with that of a commercially available sanitary napkin commonly used in India, serving as a benchmark for evaluating their practical applicability.

2 Materials and Methods

2.1 Materials

Sodium carboxymethyl cellulose (Viscosity 400 cP, MW = 2,50,000 Da) and citric acid (MW = 192 Da) were purchased from LOBA Chemicals, Chennai, India. Polyvinyl alcohol (MW = 1,25,000 Da) was purchased from Royal Scientific, Chennai, India. The sanitary napkin was procured from a domestic shop. All characterisations on test samples were performed in triplicate (n = 3). The mean of three replicates is used to express the experimental results. Statistical analyses such as the T-test and ANOVA (Analysis of Variance) are performed.

2.2 Preparation of Polymer Solution

A 10 % (w/v) Na CMC solution and a 10 % PVA (w/v) solution were prepared separately. The Na CMC solution was subjected to continuous stirring at 1750 rpm for 4 h at a temperature of 60 ^oC, while the PVA solution was stirred at 1500 rpm for 2 h at 70 ^oC. Then, these solutions were mixed with a volumetric ratio of 7:93 (Na CMC/PVA) and stirred continuously at 1000 rpm for 1 h at 60 ^oC. Na CMC has a higher molecular weight than PVA, which affects fibre formation if its volumetric proportion exceeds 7. As a result, the ratio is maintained at 7:93 during the electrospinning process[14]. Then, various concentrations of citric acid (0 %, 2 %, 4 %, 6 %, 8 %, and 10 % (w/v)) were added to the Na CMC/PVA mixture individually and stirred at 600 rpm for 12 h at 30 ^oC. Table 1 provides specific details of the test samples.

2.3 Electrospinning of Na CMC/PVA/CA Solution

A lab-scale horizontal electrospinning apparatus (Gamma High Voltage Electrospinning Machine Pvt. Ltd., India) was used to develop the fibrous web. The apparatus consisted of main elements such as a high voltage supplier, a capillary tube with a small diameter needle (21 gauge needle), and a metallic collector. To commence the spinning process, 5 mL of Na CMC/PVA/CA solution was taken in the syringe without any air bubbles and placed in the apparatus. The spinning process parameters, namely voltage, needle tip to collector distance, and solution flow rate, were set at 18 kV, 15 cm, and 0.8 mL/h, respectively. Electrospinning was carried out under a controlled environment at 27 ^oC and 60 % relative humidity (RH meter-Zeal England).

2.4 Crosslinking of Na CMC/PVA/CA Fibrous Web

After electrospinning, the resulting fibrous webs were transferred to a curing chamber (R.B. Electronic & Engineering Pvt. Ltd, Mumbai) for crosslinking reaction. The samples were pre-dried under UV light and then cured at 140 ^oC for 5 min with a material speed of 18 cm/min. Following the curing process, all test samples were conditioned at 65 % RH and 27 °C for 24 h.

Table 1 — Specific details of test samples

Sample Code	Description
CA0 (non-crosslinked)	10 % Na CMC/10 % PVA/0 % CA
CA2 (crosslinked)	10 % Na CMC/10 % PVA/2 % CA
CA4 (crosslinked)	10 % Na CMC/10 % PVA/4 % CA
CA6 (crosslinked)	10 % Na CMC/10 % PVA/6 % CA
CA8 (crosslinked)	10 % Na CMC/10 % PVA/8 % CA
CA10 (crosslinked)	10 % Na CMC/10 % PVA/10 % CA
CS	Absorbent core layer of commercial napkin (wood pulp fibre and SAP)

2.5 Surface Morphology Analysis

The morphology of electrospun samples was characterised using a scanning electron microscope (VEGA3 TESCAN SEM). A 1 cm × 1 cm sample was carefully cut, sputter-coated with gold particles, and taken for analysis. The Aztec and Inca image software were used to take the SEM images of samples.

2.6 Fourier Transform Infrared Spectroscopy

The qualitative analysis of functional groups in the fibrous web was performed using an FTIR spectrophotometer (Thermo Nicolet 6700 FTIR spectrophotometer). The IR spectrum was recorded in transmittance mode between 600 cm^{-1} and 4000 cm^{-1}.

2.7 Free Absorption Capacity (FAC) Analysis

This test was performed to evaluate the absorbency of the sample under free swelling condition[12]. It was done based on the gravimetric principle, i.e., measuring the weight change of samples after treatment with test liquids such as distilled water, 0.9 % (w/v) saline solution, and synthetic blood[17]. The saline solution was prepared by dissolving 0.9 g of NaCl in 100 mL of distilled water and stirring for 2 h at 30 °C. The synthetic blood was prepared as per the ASTM F1670 standard. A known sample weight was placed inside a nylon mesh bag with the dimensions of 6 cm x 7 cm (300 meshes per square inch) and immersed in a beaker containing 100 mL of test liquid. In the case of a commercial sample, the absorbent core layer was separated, weighed, and then placed in the mesh bag for analysis. The mesh bags, with and without samples, were immersed for 5 s, and then hung for 5 to 10 min to drain excess liquid before being weighed. Then, the FAC% was calculated using the following equation (NWSP 240.0. R2 standard).

$$\text{Free Absorption capacity (\%)} = \left(\frac{W_{WS}-W_{DS}}{W_{DS}}\right) \times 100 \qquad \ldots (1)$$

where W_{WS} is the weight of wet sample; W_{DS}, dry weight of sample; W_{WS}, difference between the weight of the wet bag containing the sample and the weight of the wet blank bag.

2.8 Liquid Retention Capacity (LRC) Analysis

This assessment was performed in accordance with the test standard NWSP 241.0. R2. In this test, 0.18 g of the sample was placed in a nylon mesh bag and immersed in the test liquids following the procedure described in the FAC test. After measuring the wet weight of the sample, it was placed in the centrifuge (REMI-R 4C-Laboratory Centrifuge) with the speed and time set at 1400 rpm and 3 min, respectively. Then, the sample was weighed to calculate its liquid retention capacity using the following equation[17]:

$$\text{Retention capacity (\%)} = \left(\frac{C_{WST}-C_{WBT}}{A_{WST}-A_{WBT}}\right) \times 100 \qquad \ldots (2)$$

where C_{WST} is the weight of centrifuged wet bag with sample; C_{WBT}, weight of centrifuged wet bag without sample; A_{WST}, weight of wet bag with sample; A_{WBT}, weight of wet bag without sample.

2.9 Absorption Under Load (AUL) Analysis

The AUL test was done to determine the absorption capacity of the sample when the compressive load is applied. This test was conducted according to the NWSP 242.0.R2 standard. Initially, the test sample was placed inside a nylon mesh bag and kept in a petri dish partially filled with test liquid. Then, a known load was placed on the sample. For saline and synthetic blood solutions, loads of 50 g/cm^2 and 3.4 Kg/cm^2 were applied[12,18]. After the test, the sample was left hanging freely in the air for 5 min and then weighed to calculate the AUL value using the following equation:

$$\text{AUL(g/g)} = \left(\frac{W_{WS}-W_{DS}}{W_{DS}}\right) \qquad \ldots (3)$$

where W_{WS} is the weight of wet sample; W_{DS}, initial dry weight of sample.

2.10 Structural Stability Test

The structural stability test for crosslinked and non-crosslinked electrospun samples was carried out based on the gravimetric principle in distilled water. The CA4 showed the highest liquid absorbency and retention percentage among all crosslinked samples and was exclusively tested for structural stability. Nylon mesh bag containing a sample and an empty bag were immersed in 100 mL of distilled water for 1 h. After immersion, the bags were removed and allowed to hang freely for 10 min to drain excess liquids from their structure. Then, they were dried at 80 ^{o}C for 20 min in a hot-air oven. After that, the weights of these samples were noted, and the structural stability was calculated using the following equation[19].

$$\text{Weight loss (\%)} = \left(\frac{W_1-W_2}{W_1}\right) \times 100 \qquad \ldots (4)$$

where W_1 is the initial dry weight of sample; W_2, final weight of sample (after oven drying the wet sample).

3 Results and Discussion

3.1 Surface Morphology Analysis

Figure 1 shows the SEM images of non-crosslinked (a) and crosslinked electrospun samples (b, c, d, e, and f), emphasising the effect of CA concentration on fibre morphology. The fibre formation is observed in both samples; however, their morphology varies depending on the CA concentrations. Continuous and uniform fibres are observed at lower CA concentrations (CA2 and CA4). However, concentrations above 4 % reduce solution stretchability, leading to bead formation (CA6) in the fibrous web[20]. Moreover, fused fibrous structures are observed at higher concentrations (CA8 and CA10). This is due to insufficient solvent evaporation[21] from the solution, leaving trapped solvent that diffuses after collection, causing interconnected junctions at the fibre contact points and resulting in a fused fibrous web[22-26].

3.2 Fourier Transform Infrared Spectroscopy

Figure 2 shows the FTIR spectrum of Na CMC, PVA, and both non-crosslinked and crosslinked samples. The crosslinked samples CA4 and CA10 are only selected for comparison to study the structural changes (bond formation) in samples with high and low absorption capacities. The sharp peaks at 1585 cm^{-1} and 3313 cm^{-1} correspond to the carboxylate groups in Na CMC and hydroxyl groups in both PVA and Na CMC. The peak at 2945 cm^{-1} confirms the presence of CH groups in Na CMC, PVA, and their blends. Furthermore, 1729 cm^{-1} and 1212 cm^{-1} peaks indicate C=O and ester bonds in both the non-crosslinked[27] and crosslinked samples[28]. However, the intensity of the ester bond peak increases in CA4 and CA10 than in CA0. It clearly shows that the addition of CA in Na CMC/PVA solution enables the formation of ester bonds that connect PVA to PVA, Na CMC to Na CMC and PVA to Na CMC[29-31]. This reaction is shown in Fig. 3.

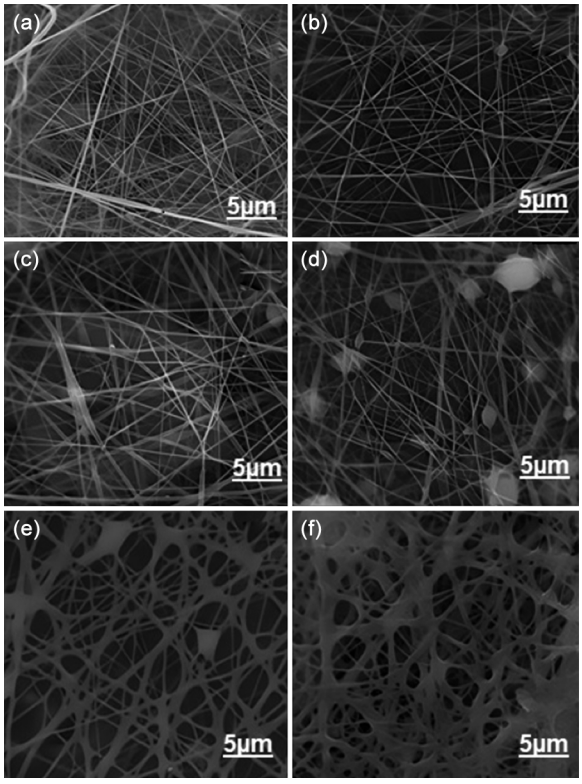

Fig. 1 — SEM images of (a) non-cross-linked sample (CA0); crosslinked samples (b) CA2, (c) CA4, (d) CA6, (e) CA8 and (f) CA10

3.3 Free Absorption Capacity (FAC) Analysis

Figures 4 (a), (b), and (c) show the free absorption percentages of samples in distilled water, saline solution, and synthetic blood solution, respectively. The figure clearly shows the effect of CA concentration on the absorption behaviour of samples.

From Fig. 4(a), it can be observed that absorption percentage increases with the increase in CA concentration up to 4 % (CA0-1608 %, CA2-1667 %, and CA4-2986 %), and then it decreases (CA6-1793 %, CA8-1229 % and CA10- 894 %) upon the further increase in CA concentration. The crosslinked samples exhibit a higher liquid absorption percentage than the non-crosslinked sample. This is due to the increase in COO^- groups quantity in Na CMC/PVA

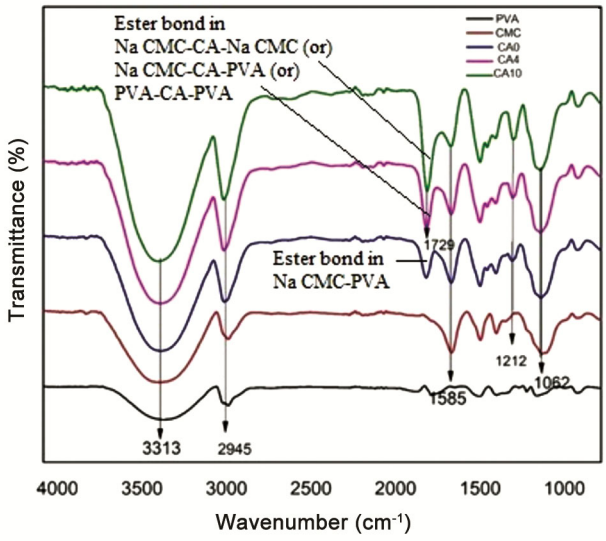

Fig. 2 — FTIR image of pure PVA, Na CMC, non-crosslinked (CA0) and crosslinked electrospun samples (CA4 and CA10)

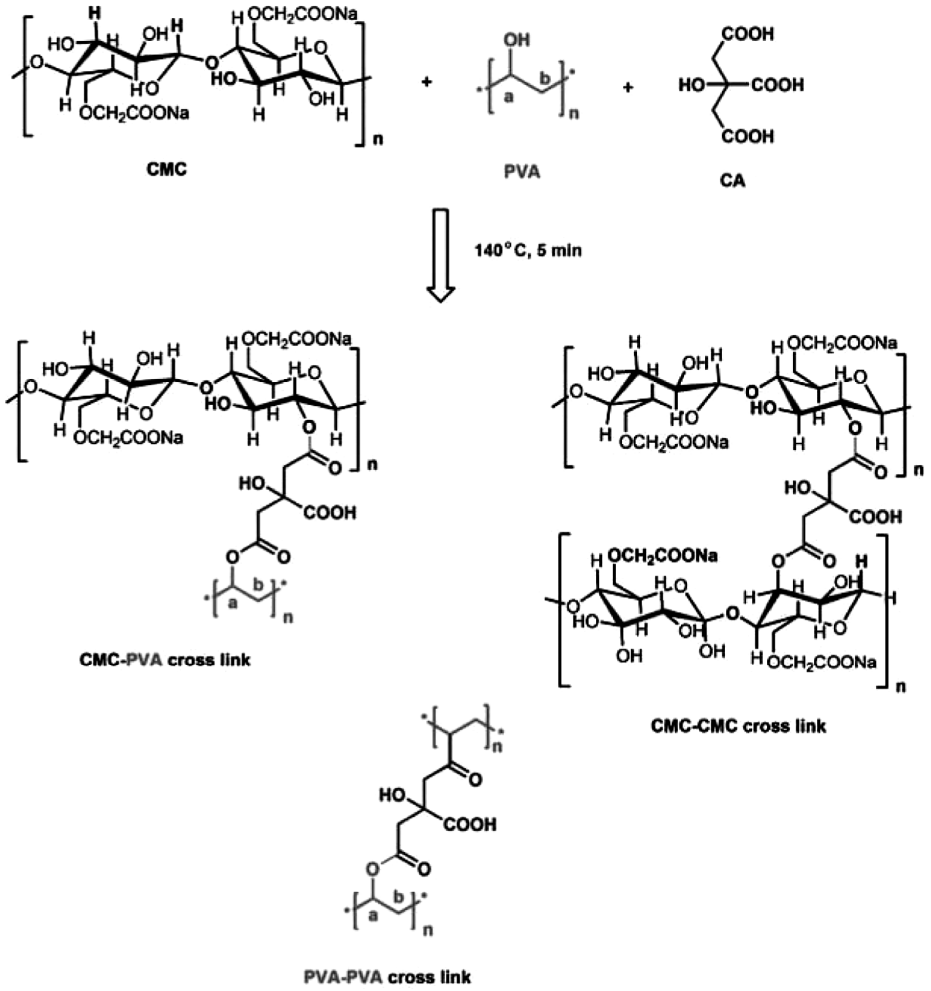

Fig. 3 — Crosslinking reaction of polymers[29, 30]

mixture after adding CA[32,33]. Interestingly, CA4 exhibits maximum free absorbency among the crosslinked samples compared to CA6, CA8, and CA10, despite the latter containing more COO^- groups. At lower CA concentrations, predominantly in CA4, the polymer structure forms crosslinking points that allow it to expand and absorb more water molecules. However, at higher CA concentrations (CA6, CA8, and CA10), the increased crosslinking points hinder the polymer structure from swelling further, reducing its liquid absorption capacity. Additionally, up to 4 % CA, unbound free hydroxyl groups in Na CMC/PVA interact with water molecules, thereby increasing the percent absorbency. However, when the CA concentration surpasses 4 %, most free hydroxyl groups form ester bonds with CA, reducing their interaction with the water molecules and lowering the absorption percentage[34,35]. The free absorption percentage of CA4 was 46.37 %, 44.17 %, 39.95 %, 58.84 %, and 70.05 % higher than CA0, CA2, CA6, CA8 and CA10 in distilled water. Furthermore, the CA4 has 43.65 % greater water absorbency than the commercial sample (CS). This difference may be attributed to the presence of a large diameter (28-30 μm) cellulosic micro fibre with a smaller amount of SAP, the exact composition of which is unknown. The reduced absorption percentage of CS may also be influenced by the possible presence of additives. A previous study has also reported similar findings[12]. Moreover, the structural transformation of superabsorbent polymers can explain a gradual increase in FAC up to 20 seconds, followed by a rapid rise until 120 seconds and then a plateau, as shown in Figure 4(a). As they absorb liquid, the polymer structures change from a glassy state to a rubbery state due to the expansion of their polymeric chain network[36].

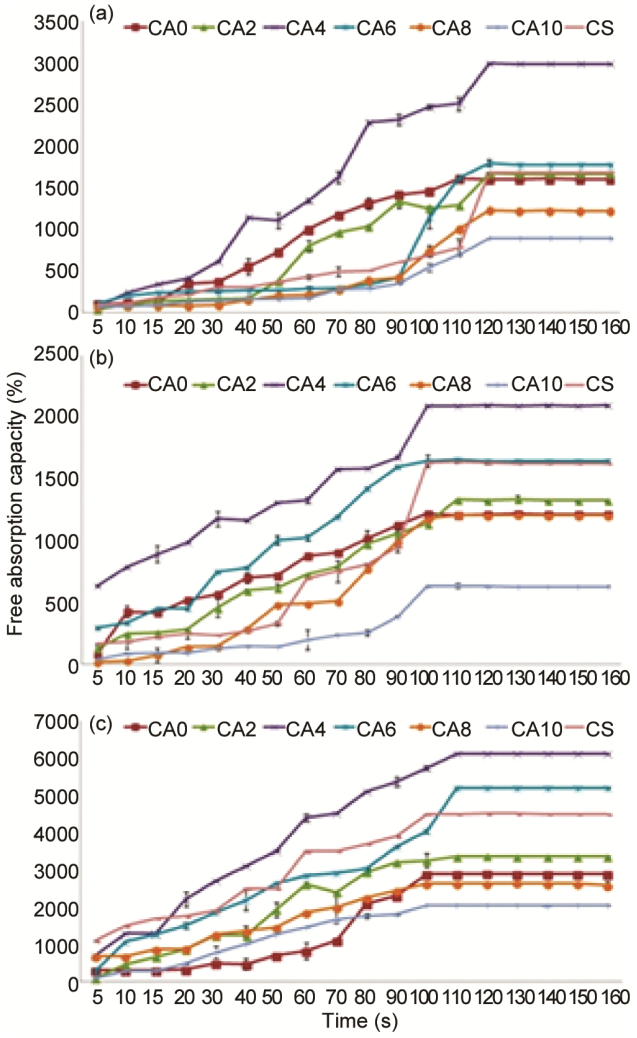

Fig. 4 — Free absorption capacity of electrospun samples and commercial napkin in (a) distilled water, (b) saline solution and (c) synthetic blood (two-way ANOVA-significant difference is found between the samples with p-value < 0.05)

Figures 4(b) and (c) show the effect of CA concentration on the free absorption capacity of test samples in saline and synthetic blood solutions. The graphs for these test solutions show a similar trend for distilled water in Fig. 4(a). However, the samples tested in saline solution exhibit lower FAC than those in distilled water and synthetic blood solution, which may be due to the neutralisation of counter ions (Na^+ and COO^-) in Na CMC by the counter ions in the saline solution (Na^+ and Cl^-)[37]. The FAC of CA4 is observed to be 41.96 %, 36.2 %, 21.36 %, 42.33 %, and 70.01 % higher than CA0, CA2, CA6, CA8 and CA10 in saline solution. Further, the CA4 has 22.627% better saline absorbency compared to CS. The percent free absorbency of test samples in the

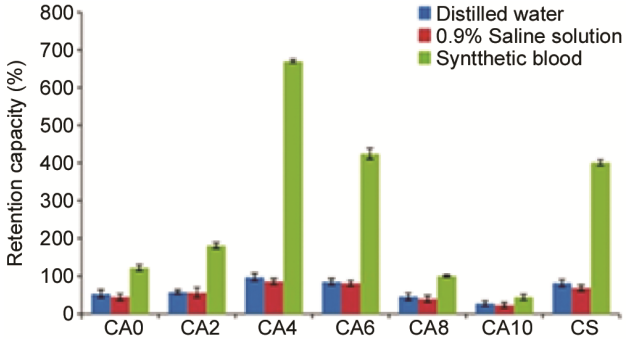

Fig. 5 — Liquid retention capacity of electrospun samples and commercial napkin in distilled water, saline solution, and synthetic blood (Two-way ANOVA-significant difference is found between the samples with p-value < 0.05)

synthetic blood solution is higher than in saline and water. The FAC of CA4 is observed to be 52.42 %, 44.90 %, 14.88 %, 56.68 %, 66.34 % and 26.19 % higher than CA0, CA2, CA6, CA8, CA10 and CS in synthetic blood.

3.4 Liquid Retention Capacity (LRC) Analysis

Figure 5 illustrates the effect of CA concentration on the liquid retention capacity of samples in distilled water, saline, and synthetic blood solution. The graph shows that the crosslinked samples have a higher liquid retention percentage than the CA0. In general, the formation of crosslinking points in the SAP structure helps to retain the liquid molecules against the force applied to the samples during the test. Hence, the formation of ester bonds in the Na CMC/PVA/CA polymer network could be the reason for retaining the test liquids against the applied force. The CA4 showed a higher liquid retention percentage in all test liquids than the other samples. The lowest liquid retention percentage is observed in CA2 and CA10 samples due to the formation of lower and higher amounts of crosslinking points. This result confirmed the role of CA concentration in the sample's liquid retention property.

Furthermore, it is observed that the nature of the solution also influences the samples' retention capacity. The former exhibits a lower value when comparing the retention percentage of samples in saline and distilled water, as seen in the FAC analysis[38,39]. However, the LRC of samples tested in synthetic blood solution is higher than in distilled water and saline solution. The LRC of CA4 is observed to be 16.8 %, 19.8 %, and 40.17 % higher than CS in distilled water, saline, and synthetic blood, respectively.

Table 2 — Structural stability of non-crosslinked and crosslinked electrospun fibrous web*

Sample code	Initial weight, mg	Final weight, mg	Weight loss, %
CA0	20	15.3	24
CA4	20	19.6	2

*T-test analysis: significant difference is found between two samples with a p-value of 0.000291< 0.05)

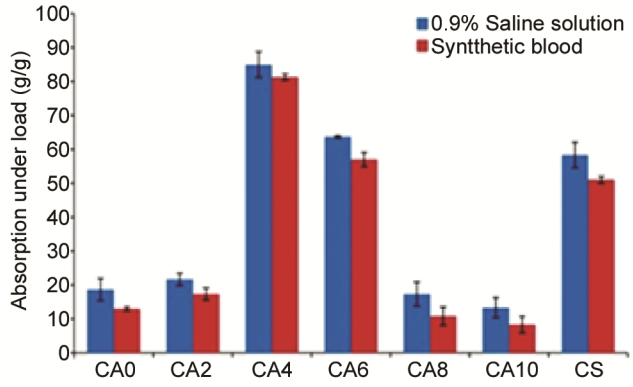

Fig. 6 — Absorption under load value of electrospun samples and commercial napkin in salinesolution and synthetic blood (two-way ANOVA-significant difference is found between the samples with p-value < 0.05)

3.5 Absorption Under Load (AUL) Analysis

This test investigates the effect of applied load on the swelling behaviour of the sample, which is crucial for evaluating the potential use of samples in napkin application. Figure 6 illustrates the impact of CA concentration on the AUL value of test samples in saline and synthetic blood solution. As in the case of FAC and LRC, the CA4 show a higher AUL value than other samples. The AUL value of CA4 in saline is 79.57 %, 81.48 %, 84.43 %, and 29.7 % higher than CA0, CA8, CA10, and CS, respectively. Similarly, the AUL value of CA4 in synthetic blood solution is 84.72 %, 87.89 %, 93.50 %, and 30.40 % higher than CA0, CA8, CA10, and CS, respectively. Further, unlike in LRC, the AUL value of test samples in saline solution is higher than in synthetic blood solution. This may be due to the presence of a thickening agent in the synthetic blood solution, which hindered its diffusion into the fibrous web structure.

3.6 Structural Stability Test

This test has been conducted on non-crosslinked (CA0) and crosslinked samples (CA4). However, it is performed exclusively on CA4, exhibiting maximum liquid absorption and retention capacities among all the crosslinked samples. In general, Na CMC and PVA polymers are water-soluble. However, due to CA, the formation of crosslinks in the polymeric structure restricts their solubility in distilled water. Furthermore, Table 2 presents the weight loss percentage of CA0 and CA4 before and after the treatment with distilled water. The weight loss percentages of CA0 and CA4 are around 24% and 2%, respectively. This may be due to the absence or poor formation of crosslinking points in CA0, which is responsible for the higher weight loss compared to CA4. The minimum weight loss percentage observed in CA4 may be attributed to the presence of unreacted and loosely bound low molecular weight polymers.

4 Conclusion

This study describes the systematic procedure for fabricating a superabsorbent fibrous web composed of Na CMC/PVA/CA using the electrospinning technique. This study concludes the influence of CA concentration on morphological, free liquid absorption, liquid retention, and absorption under load properties of electrospun samples. The SEM pictures confirm the changes in the morphology of electrospun samples as the CA concentrations are varied beyond 4 %. In all tests, the CA4 sample shows higher free absorbency, liquid retention capacity, and AUL value than the CA2, CA6, CA8, CA10, and CS samples. The structural stability test reveals that the crosslinked sample (CA4) exhibits higher resistance to water solubility than the non-crosslinked sample (CA0). Therefore, due to its high absorbency, structural stability and the use of environmentally compatible polymers, the electrospun sample (CA4) could serve as a potential biodegradable superabsorbent material for hygiene absorbent applications.

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