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RESEARCH ARTICLE

Sustainable Superabsorbent Polymer Synthesis from Pakhuri Leaves and Wheat Husk for Incontinence Pads

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Abstract

Carboxymethyl cellulose (CMC) was synthesized from Pakhuri (*Ficus glaberrima* Blume) and Wheat (*Triticum aestivum* L) husk, two abundant agro-forest wastes in Nepal, to develop sustainable superabsorbent polymer (SAP) precursors for incontinence pad and diapers applications. The synthesized CMC exhibited degrees of substitution (DS) in the range of 1–2, confirming successful etherification. However, CMC yields (40–65%) and swelling capacities (~200%) were relatively low compared to commercial SAPs. Characterizations (XRD, FTIR, and Brookfield's viscosity measurement) revealed that Pakhuri has relatively superior SAP quality for incontinence pads and baby diapers since Pakhuri SAP is substitution efficient with low viscosity. However, substitution alone is insufficient to ensure high absorbency, with crosslink density, residual salts, and polymer morphology playing key roles in limiting water uptake. Despite these limitations, the study demonstrates the feasibility of converting underutilized biomass into functional SAP precursors. Future optimization of crosslinking, purification, and porosity enhancement is expected to significantly improve swelling performance, offering a pathway toward sustainable, low-cost SAPs suitable for hygiene and healthcare applications.

Keywords: Absorbency; Carboxymethyl cellulose; Degree of substitution; Incontinence pads; Superabsorbent polymer

Introduction

The issues of sanitary pad accessibility have garnered significant attention in Nepal. This challenge presents an opportunity to explore sustainable solutions utilizing locally available green resources. The issue can be effectively addressed by utilizing local plant resources with appropriate chemical modifications. Nepal produces a substantial volume of plant waste and by-products from forests, farming, and agro-based industries, much of which is commonly seen littering the environment. However, this challenge can be transformed into an opportunity by converting plant wastes into valuable products such as superabsorbent polymers (SAPs). Forest and agricultural by-products that are often discarded can be efficiently utilized to produce these useful SAP materials. This approach not only prevents the wastage of agro-forest resources but also adds value to them [1], ultimately boosting both the local and national economy. Research records and findings

indicate that only limited efforts have been made on this issue in Nepal, despite significant potential to replace existing acrylic-based SAPs with plant-based alternatives.

SAPs possess a three-dimensional hydrophilic network with a moderate level of cross-linking, enabling them to absorb and retain substantial amounts of water—often 1000 to 1500 times their dry weight—without dissolving even under applied pressure [2]. These materials surpass conventional hydrophilic substances in both water absorption and retention capacities, continuing to function effectively under load [3-6]. When dry, the polymer chains are coiled; upon absorbing liquid, the chains uncoil, causing the network to expand. The absorbed liquid is then trapped within the gaps of the molecular network, forming a gel that securely holds the fluid [7,8]. This process is driven by osmotic pressure differences, consistent with Flory's network theory, which explains the swelling and absorption dynamics [9]. This exceptional water absorption capacity is attributed to functional groups such as carboxylic acids, partially neutralized carboxylates, carboxylate salts, and carboxamides [10-14]. Owing to these properties, SAPs find extensive applications across various fields, including hygiene products like diapers, soil moisture retention in agriculture, and medical devices [15-18].
The SAPs are extensively used in disposable diapers, adult incontinence products, and feminine hygiene products to absorb and lock away moisture, keeping the user dry and comfortable. Such SAPs, often made from materials like Carboxymethyl Cellulose (CMC) is an anionic linear polysaccharide [19,20]. The CMC is indeed a versatile modification of cellulose present in almost 90 % of plants or plants wastes. Different plant species contain varying amounts and types of cellulose, hemicellulose, and lignin. Cellulose is a polysaccharide made up of anhydro-glucose repeating units connected by β-1,4- glycosidic bonds. Hence CMC possesses numerous hydroxyl and carboxylic groups. Such structure gives CMC its mechanical strength and allows for the tuning of properties like hydrophilicity, viscosity, and other properties [21,22]. The CMC prepared from plant resources is highly viscous, nontoxic, non-allergenic, and biodegradable. The hydroxyl and carboxylic groups in such CMC allow it to bind, absorb and hold water. The effectiveness of CMC in its various applications depends on factors like purity, degree of polymerization (DP), degree of substitution (DS), and uniformity. These factors can be controlled during the manufacturing process to tailor CMC for specific purposes.

Several methods have been suggested for the extraction of cellulose from plant materials. These methods include chemical, mechanical, and enzymatic processes. Synthesis and tuning of SAPs properties can be done by fixing CMC, NaOH as well as HCl concentrations along with cross-linker and neutralizing agents e. g. pH, temperature etc. [10, 23, 24]. Synthesis of a novel SAP has been reported by Chen *et. al* [25] through graft copolymerization of sodium acrylate and 1-vinyl-2-pyrrolidone onto the chain of NO-carboxymethyl chitosan which is found to absorb 1268 g/g water, over 165 g/g normal saline. Likewise, radiation induced grafting of acyl amide (AM) onto CMC in the presence of N, N-methylene bisacrylamide (MBA) as cross-linker has been reported by Hemvichian *et. al* [26]. In this regard, Fang *et. al* [27] reported a new biodegradable polymer by fabrication in rice starch. The choice of method depends on factors such as the plant source, intended application, and environmental considerations.

The synthesis of CMC from green resources like *Ficus glaberrima* Blume (i.e. Pakhuri in Nepali) leaves and *Triticum aestivum* L. (i.e. Wheat) husk and study of their properties are,

of course, novel and sparsely documented in the literature. Hence, this study aims to introduce an alternative resource for producing CMC from Pakhuri leaves, an unused biomass, as well as wheat husk to replace the acrylic based SAPs. The study includes the characterization of CMC, focusing on its yield, degree of substitution (DS), functional groups, viscosity, and absorbency. These properties are evaluated for their potential applications in feminine hygiene products, adult incontinence pads, baby diapers, and surgical dressings. This would mitigate the environmental impact of diapers by improving the biodegradability of the synthetic components found in the present existing marketplace.

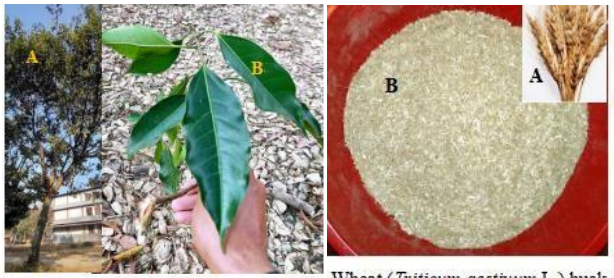
Materials and Methods

This is an experimental study, where the researchers undertook the collection and identification of Pakhuri plant (leaves) and Wheat husk for cellulose extraction (**Table 1**).

Table1: Taxonomic classification of the plants used in this study

Pakhuri (Nepali, local name)	Wheat (Common or bread wheat)
Kingdom: Plantae (Plants) **Division:** Magnoliophyta (Flowering plants) **Clades:** Magnoliopsida (Eudicots) **Order:** Rosales **Family:** Moraceae (Mulberry family) **Genus:** Ficus (Fig trees) **Species:** *Ficus glaberrima*	**Kingdom**: Plantae (Plants) **Phylum**:Tracheoph (Vascular plants) **Clades**: Liliopsida **(Monocots)** **Order**: Poales (A group of grasses and sedges) **Family**: Poaceae (grass family) **Genus**: Triticum **Species**: *Triticu m aestivum L.*

The Pakhuri plant leaves were collected from the spinney of Prithvi Narayan Campus, Pokhara, and the Wheat husk was collected from Dhangadhimai-10, Siraha, Nepal (**Figure 1)**. Both the Pakhuri leaves and Wheat husk were dried well in the sun light and then ground by using a grinder machine (Baltra 750 W Turbo) into powder form. The powders were sieved using a standard test sieve shaker (Indian make), with mesh sizes of 60, 75, 90, 100, and 150. For international comparability, these mesh sizes were expressed in terms of their equivalent nominal opening sizes according to American Society for Testing and Materials (ASTM) E11 standards, corresponding to approximately 250, 200, 167, 149, and 100 µm, respectively. These sieved mesh of various sizes were subjected to further treatments with reagents and solutions. Various concentrations of NaOH and Monochloro-acetic acid (MCA) were prepared (Ref table-2) by following standard protocols of calculations and solution preparation. These sample meshes were subjected to extraction of cellulose and CMC starting with delignification. The chemicals used were of analytical reagent (AR) grade and the experimental procedures followed and adopted were as per the standard protocols.



Pakhuri (*Ficus glaberrima* Blume) **A**-Tree, **B**-Leaves

Wheat (*Triticum aestivum* L.) husk **A**-wheat with stalk, **B**-wheat husk

Figure1: Experimental plant parts employed for SAPs preparation

Delignification

This was carried out starting from the pre-treatment of the samples with acetic acid and 4% sodium chlorite ($NaClO_2$) keeping the sample contents in water bath for two hours at 70°C followed by filtration and washing with cold water. All the samples were then subjected to bleaching and hydrolysis. This

involves treating the samples with 4% sodium hypochlorite (NaOCl) and 2 % NaOH solution in water bath maintained at 30°C for half an hour. Then these were shaken for 2h using type WA- 200 flask shaker at speed of 800 Osc/min. All the samples were then filtered, the residues were washed with cold water continuously and tested until pH 7 (approx.). The absence of lignin *i.e.* complete removal from the sample was tested by treating with Phloroglucinol-HCl (red color test). The biomass thus obtained is chlorite-cellulose which was dried in an oven at 60°C for 2h.

Cellulose Separation

The dried chlorite-cellulose was treated with 18 % NaOH for 2h. The cellulose was separated by filtration which was first washed with 2% acetic acid and then with hot distilled water (28).

Carboxymethylation

The cellulose samples thus obtained was subjected to *alkalization* and then *etherification.* Alkalization involves treatment of the samples with 10% NaOH and pure Isopropanol, shaking them for 2h using flask shaker. The samples were then filtered and washed with a solution of acetic acid (10%) and methanol (70%). *Etherification* involves treatment of the samples with various reagents as mentioned in the Table-2. Mesh sizes 60, 75, 90, 100, and 150 micron each went through the T_1, T_2, T_3 and T_4 experimental sets. As shown in the table, only one parameter out of four (indicated by bold letters) was changed in each set of the etherification experiment. For example, in the set T-1, concentration of NaOH was varied as 2M, 4M, 6M, 8M to 10M for each mesh size. Likewise, in the set T-2, time was varied, in the set T-3 temperature was varied and in the set T-4 MCA concentration was varied.

Alkalization (mercerization)

$$\text{Cellulose–OH} + \text{NaOH} \longrightarrow \text{Cellulose–ONa}^+ + H_2O$$

Etherification (Carboxymethylation)

$$\text{Cellulose–O-Na}^+ + \text{Cl–CH}_2\text{–COONa} \longrightarrow \text{Cellulose–O–CH}_2\text{–COONa} + \text{NaCl}$$

Table 2: Etherification brief, showing four sets with variation in the reaction parameters (T-1: NaOH concentration variation, T-2: Time variation, T-3: Temperature variation and T-4: MCA concentration variation) without any cross-linker)

Expt. Set	**NaOH** [M]	**MCA** [M]	**Time** (h)	**Temp.** (°C)
T-1	2, 4, 6, 8, 10	1	0.5	65
T-2	10	1	1,1.5, 2,2.5,3	65
T-3	10	1	0.5	50,60, 70,80, 90
T-4	10	0.5,1,1.5, 2, 2.5	0.5	65

Characterization Method

The carboxymethyl cellulose (CMC) yield from each sample has been calculated using the following formula [29].

$$\text{CMC Yield, \%} = \frac{\text{Wt. of obtained CMC}}{\text{Wt. of dried Cellulose}} \times 100$$

Degree of Substitution (DS) value has been calculated using following formula; [3-8]

$$\text{D.S.} = \frac{162 \times \%\text{CM}}{[5800 - (57 \times \%\text{CM})]}$$

Water Absorption Capacity (WAC) has been measured using tea-bag method according to the following equation [9-13]

$$\text{WAC} = \frac{W_1 - W_0}{W_0}$$

Where W_0: dry weight and W_1 wet weight

IR Tracer-100 (Shimadzu) FTIR spectrometer was used for the identification of the CMC using in the wavenumber range of 500-4000 cm^{-1}.

XRD analysis of sample were carried out by using HighScore (CuKα radiation of wavelength λ = 1.5406 Å) with 18kW rotating anode-based powder diffractometer. The data were collected

in continuous scan mode in 2θ range of 5 – 90 degrees at a scan rate of two degrees per minute and a step interval of 0.02 degrees.

Results and Discussion

The FTIR analysis and Phloroglucinol-HCl test are highly reliable tests to confirm the status of lignin in the plant based cellulose. The absence of peaks at 1510 cm^{-1}(aromatic C=C stretch) and 1600 cm^{-1} (aromatic ring vibration) confirm the removal of lignin. The reaction mixture was filtered which gave approximately no residue ensuring almost complete removal of the lignin. Hydrophilic groups like –COOH, -OH, -$CONH_2$ etc. are critical in SAPs for absorption performance of liquids. The O-H stretching showing broad peaks around 3200-3600 cm^{-1} indicate H-bonding. Peaks near 1700 cm^{-1} represent is C=O stretching which represents –COOH group. Peaks around 1000-1200 cm^{-1} is C-O stretching which correspond to ether or amide bonds. The partially neutralized carboxylic groups (e.g., -COONa) enhance absorbency. The peaks in the 1400–1450 cm^{-1} range suggest the presence of carboxylate salts. The appearance of new peaks or a decrease in intensity of original peaks signifies structural changes in the polymer. It is obvious from the spectra that the cellulose has undergone caroxymethylation. The FTIR (**Figure 2**) both (a) Pakhuri and (b) Wheat husk samples clearly show the presence of N-H, O-H, C-H as well as C=O functional groups. These functional groups play important role in the absorbency property of the SAPs applicable in incontinence pads.

Figure 3 shows the XRD pattern of CMC synthesized from Pakhuri (*Ficus glaberrima* Blume) leaves and that of Wheat (*Triticum aestivum* L) husk. The XRD patterns suggest that CMC from Pakhuri is mostly amorphous material with some degree of short-range order likely due to cellulose derivatives. The broad peak at 2θ~20° is consistent with polysaccharide-based superabsorbent materials (JCPDS 03-0226), confirming its plant-derived nature. It is obvious that CMC is not completely amorphous, but have a relatively high degree of crystallinity. The gradual decline in intensity beyond 30° suggests disordered molecular arrangements, confirming an amorphous nature. This structure is advantageous for water absorption and swelling properties, making it suitable for biodegradable absorbent applications. Likewise, the XRD pattern of SAP synthesized from Wheat (*Triticum aestivum* L) husk also shows a broad peak at ~20°.

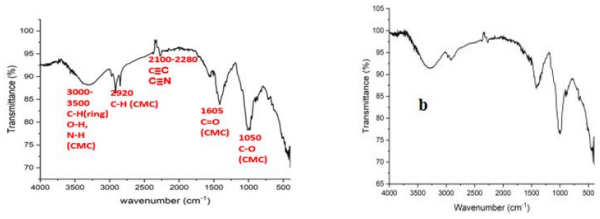


Figure 2: FTIR (a) CMC of Pakhuri leaves (P-150) (b) CMC of Wheat husk (W-60)

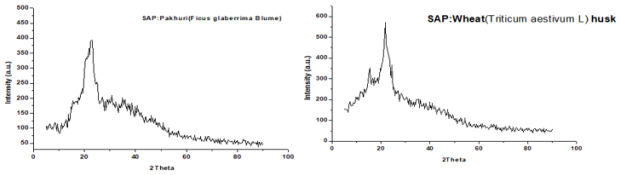


Figure 3: Powder XRD pattern of CMC from Pakhuri leaves (P-60) and Wheat husk (W-60)

However, the peak intensity is higher in this case, which may indicate a slightly higher degree of crystallinity, possibly due to differences in the cellulose or hemicellulose content of wheat husk. The lack of sharp peaks beyond 30° confirms the absence of highly crystalline components such as inorganic salts or structured polymers. The gradual decline suggests a complex amorphous matrix that is advantageous for water absorption and swelling properties, making it an eco-friendly alternative to synthetic superabsorbents.

Effects of change in concentration of NaOH, and that of MCA, time and temperature have been studied to determine the CMC yield, Viscosity, Degree of substitution (DS), and absorbency of the SAP-materials. The performance of SAP is found influenced by the particle size, yield, purity of CMC, as well as by the DS values. The DS value is the primary indicator of CMC that helps to optimize the reaction conditions and defines the future applications of the SAPs. The Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction (XRD) of the SAP samples were performed. The origin plots of the CMC yield, DS, absorbency etc. are shown below (**Figure 4, 5 and 6**).

Figure 4 shows CMC (%) yields of Pakhuri leaves and Wheat husk at four experimental sets T1, T2, T3 and T4 (as described in the **Table-2**). Evidently, CMC yield (around 40%) does not vary significantly with the mesh size. Both the plant samples showed CMC yield in the range of 35-60 (approx.) % with the sequence of CMC percentage T1 >T2 >T3 >T4. Thus, T1 gave the highest CMC % because of stronger alkalization (Ref table-2) leading to higher substitution efficiency. The lowest yield is observed with T4 which suggests that simply increasing MCA concentration is not effective; beyond an optimum level, MCA leads to by-product formation (e.g. sodium glycolate), which reduced CMC yield.

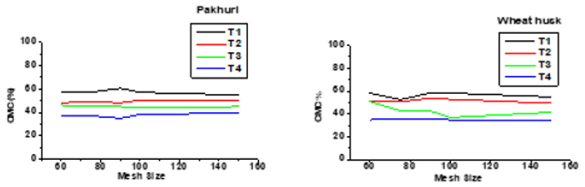


Figure 4: Plots of CMC % *vs.* Mesh size of *Ficus glaberrima* Blume (Pakhuri) leaves and *Triticum aestivum* L. (Wheat) husk after etherification at T1, T2, T3 and T4 experimental sets [Ref table-1]

Thus, NaOH concentration is the most critical factor controlling CMC synthesis from both Pakhuri and Wheat husk substrates. Smaller mesh or particle size provides a larger surface area-to-volume ratio whereby solvents (e.g. NaOH) used for hydrolysis can penetrate more effectively during pretreatment process. This enhances the removal of lignin and other non-cellulosic components resulting in higher extraction efficiency maintaining high-quality cellulose. Temperature accelerates the removal of lignin when combined with reagents like NaOH. Optimal temperatures ensure effective lignin removal without significant damage to cellulose fibers. The temperatures (~60–100°C) are usually ideal for cellulose extraction, ensuring effective removal of impurities without degrading the cellulose.

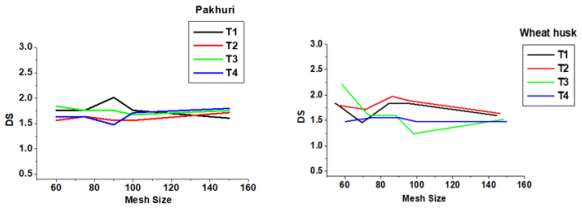


Figure 5: Plots of DS *vs.* Mesh size of *Ficus glaberrima* Blume (Pakhuri) leaves and *Triticum aestivum* L. (Wheat) husk after etherification at T1, T2, T3 and T4 experimental sets.

Figure 5 shows variation of DS with mesh size in the CMC of Pakhuri leaves and wheat husk. Evidently, both the plants samples exhibited DS value higher than 1.5 which is, in general, higher than the DS (0.6 -1.0) of SAPs employed in the incontinence pads of the present market [13]. DS value greatly affects the CMC properties. Higher DS values indicate a higher incorporation of the functional groups e.g. –COOH, -OH, or –CO within the polymer network. These groups significantly increase the hydrophilicity of SAPs, allowing them to absorb and retain more liquids or aqueous solutions which is crucial for applications in hygiene products like sanitary pad and diapers where high absorption capacity is crucial. Indeed, higher DS of CMC provides better liquid retention and

reduced leakage when used as SAPs in such incontinence pads.

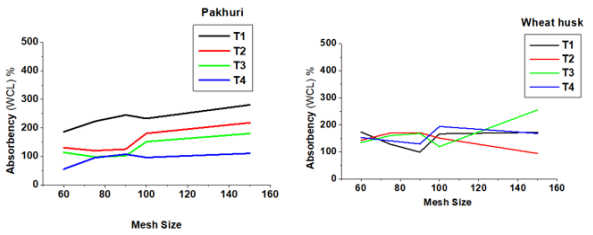


Figure 6: Plots of absorbency *vs.* mesh size of *Ficus glaberrima* Blume (Pakhuri) leaves and *Triticum aestivum* L. (Wheat) husk after etherification at T1, T2, T3 and T4 experimental sets.

In the present study, DS ≥ 1.5 for most of the SAP samples (**Figure 5**), but absorbency is still <100% (**Figure 6**) in some cases which means the product is likely over-substituted, poorly crosslinked, or too soluble. The high DS makes CMC soluble in water not behaving as cross-linked hydrogel. Cross-linked density gives a 3D network capable of retaining large amounts of water. Without good cross-linking the polymer just disperses exhibiting poor absorbency. Another possible reason may the counter-ion effect due to Na^+, Ca^{2+} etc. High DS means many carboxylate (–COONa) groups but in water with salts (NaCl, $CaCl_2$), charge screening or ionic cross-linking occurs, limiting swelling. So in saline or real-world conditions, absorbency drops dramatically.

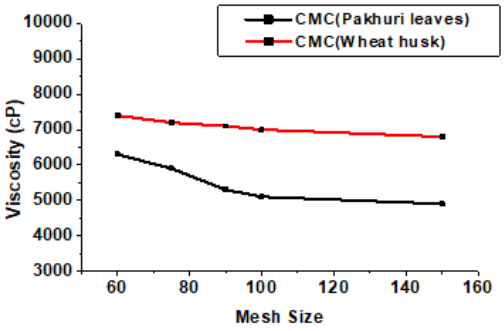


Figure 7: Plots of Viscosity *vs.* Mesh size of *Ficus glaberrima* Blume (Pakhuri) leaves and *Triticum aestivum* L. (Wheat) husk CMC measured by using Brookfield's Viscometer.

As evident from the plots of absorbency *vs.* mesh size of both the experimental plant samples (**Figure 6**) absorbency in both the cases is ≤ 100 with wheat husk showing higher absorbency at the experimental conditions T3 and T4 where T3 stands for the temperature variations (50-90°C) and T4 stands for MCA concentration variation *viz.* 0.5 M to 2.5M, (Ref table-2). Sometimes SAPs show high Free Swell Capacity (FSC) but low Absorbency Under Load (AUL). Since saline water was used to test absorbency of the prepared SAP samples in this study, the possible reasons for overall low absorbency of the SAP may be (a) residual NaCl/NaOAc/MCA (b) inappropriate cross-linking and (c) chain degradation during mercerization. **Figure 7** shows the viscosity variations of the various mesh of Pakhuri and Wheat husk CMC. Viscosity studies are very significant for characterizing CMC-based SAPs, because viscosity links the molecular structure (DS, chain length, crosslinking) to the absorbency. Commercial CMC is often sold by viscosity grades (measured in mPa.s at 2% solution, 25°C). It is evident from the Figure 7 that CMC of wheat husk has higher viscosity for all mesh sizes than that of Pakhuri leaves. It indicates that the quality or type of cellulose in them are different. The SAP with lower viscosity offers softer feel [13] hence more preferable for incontinence pads or baby diapers. Obviously, Pakhuri SAPs seems superior than wheat husk in this regard. However, in both the cases, smaller mesh (bigger particles according to ASTM E11) result in a higher viscosity for a given shear rate and solid fraction. Larger particles (retained on coarser mesh, like 60) have lower specific surface area but often contain more intact cellulose fibrils and hemicellulose fractions. These longer fibers form a physical entanglement network in

solution, which increases resistance to flow thus resulting higher viscosity. Coarser particles usually retain more of the natural cell-wall matrix, which swells more in alkaline solution during CMC preparation. This swelling enhances water binding and increases viscosity. Higher viscosities observed for coarser fractions (larger particle size) indicate greater chain entanglement and swelling ability, which favors gel integrity and enhances liquid retention capacity of SAPs. However, excessively high viscosity may hinder fluid diffusion into the polymer network, suggesting that an optimal particle size distribution is critical for maximizing both absorption rate and retention. Such CMC based SAPs with ultrafine particles may be more suitable for medical grade and incontinence pads that require rapid absorption; however, excessively fine particles may hinder processing and uniform distribution in formulations. Thus, an optimum range must be identified to avoid excessive viscosity that can lower or even prevent the functional performance of the SAPs in real-world applications [29-31].

Conclusions

This study demonstrates that Pakhuri (*Ficus glaberrima* Blume) leaves and wheat (*Triticum aestivum* L.) husk, two abundant agro-wastes, can be valorized into carboxymethyl cellulose (CMC) suitable for superabsorbent polymer (SAP) applications. DS values in the range of 1–2 confirmed successful etherification, although yields (40–65%) and swelling ratios (~200%) remained modest. The results suggest that substitution alone is insufficient to ensure high absorbency, and factors such as crosslink density, residual salts, and polymer morphology strongly influence swelling performance. Despite these limitations, this work establishes the feasibility of converting underutilized Nepalese biomass into functional SAP precursors. Future efforts focusing on optimized crosslinking, purification, and porosity control may substantially improve absorbency, paving the way for sustainable, low-cost SAPs for incontinence care and related applications.

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Author's contribution statement

S. N. Jha: Laboratory works & experimentation, characterizations and analysis, Writing-original draft, **S. Poudel, S. Kamar, S. Kamar, S. Shrestha, B. Paudel,** Laboratory works & experimentation; N. D. **Bastakoti**: Plants identification, and classification; K. Gurung: Microbiological experimentation; **S. K. Gautam**: Characterizations and analysis, Validation, Writing: review and editing; **R. J. Yadav:** Conceptualization, Methodology, XRD-Characterizations, Project admin & MS-development, Writing: review and editing

Conflict of interest

The authors do not have any conflict of interest throughout this research study.

Data availability statement

The data supporting these study findings can be made available through the corresponding authors on request

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