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Lignocellulose-based Hydrogels derived from modified *Tithonia diversifolia* pith: Synthesis and Characterisation

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ABSTRACT

This study investigated the synthesis and properties of novel hydrogels: Sunflower pith-g-polyacrylamide (SFP-g-PAm) and carboxymethyl sunflower pith-g-polyacrylamide (CMSFP-g-PAm) from native and carboxymethyl (CM) modified *Tithonia diversifolia* pith (TDP) through free radical graft copolymerisation, using potassium persulphate (KPS) as an initiator, acrylamide (Am) as a monomer, and N,N'-Methylenebisacrylamide (MBA) as a crosslinker in a nitrogen environment at 70 ºC. Hydrogel synthesis conditions were optimised by varying biomass content, KPS, and MBA, with water absorbency as the primary metric. A crucial finding of this investigation was the enhancement in water absorbency exhibited by the CMSFP-g-PAm hydrogels (152.00 g/g) compared to their SFP-g-PAm counterparts (89.33 g/g) across all optimised conditions, which were determined as 2.43 wt% KPS, 0.16 wt% MBA, and 31.4 wt% lignocellulose biomass. The CMSFP-g-PAm hydrogels demonstrated improved water retention capacity (50.03%) of its initial swollen mass after 8 h compared to 15.23% in their unmodified counterparts. Fourier transform infrared (FTIR) spectroscopy confirmed the graft polymerisation and carboxymethyl modification of the hydrogel. Scanning electron microscopy (SEM) revealed a porous network in both hydrogels, with the CM-modified sample exhibiting a larger pore structure, contributing to its superior water absorption and retention properties. This research highlights the potential of chemically modified *Tithonia diversifolia* pith as a sustainable feedstock for superabsorbent materials.

Key words: Carboxymethylation, hydrogel, *Tithonia diversifolia* pith, water absorbency

INTRODUCTION

Agriculture is fundamentally responsible for global food security, economic stability, and the livelihoods of people [1]. However, this critical sector is grappling with challenges, primarily exacerbated by the impact of climate change [2]. Erratic weather patterns, prolonged droughts, and severe land degradation are increasingly compromising crop yields and agricultural productivity worldwide, particularly in vulnerable regions [3]. There is need for innovative and sustainable agricultural practices that can mitigate these adverse effects and ensure resilient food systems.

The development of functional materials, such as superabsorbent hydrogels, offers a promising solution. These polymeric networks possess extraordinary water retention capabilities, often absorbing several times their own weight in water [4]. When incorporated into agricultural soils, they act as miniature reservoirs, gradually releasing stored water and dissolved nutrients directly to plant roots [5].

Traditionally, hydrogels have been synthesised from synthetic polymers, raising concerns about their environmental footprint. Consequently, there is a growing global imperative to shift towards "green" alternatives derived from abundant, renewable, and biodegradable natural resources [5]. Lignocellulosic biomass, the most prevalent organic material on Earth, stands out as an ideal precursor for the development of such eco-friendly hydrogels [6].

Tithonia diversifolia (TD), commonly known as the Mexican sunflower or wild sunflower, is a flowering plant found in many parts of the world, including Nigeria. While possessing a vibrant appearance, it is, however, an aggressive, pervasive and troublesome fast-growing weed across many tropical and subtropical agricultural landscapes, notably in the South-western states of Nigeria. Little information is available on the importance of this plant. However, in recent time, the use of TD stem has gained more interest and some of its reported applications include; biodegradable packaging materials, feed supplement for livestock, and biocomposites [7, 8]. The substantial biomass of *Tithonia diversifolia* often remains under-utilised or discarded, representing a significant untapped resource. Therefore, valorising this abundant weed by transforming its pith into a functional hydrogel not only addresses the environmental nuisance it poses but also aligns perfectly with circular economy principles.

This study investigates the potentials of utilising *Tithonia diversifolia* pith to synthesise a novel hydrogel using combination of acrylamide, N, N'-methelynebisacrylamide, potassium persulphate, and *Tithonia diversifolia* pith as monomer, crosslinker, initiator, and skeletal polymeric backbone (substrate) respectively.

MATERIAL AND METHODS

Materials

Acrylamide, N,N'-Methylenebisacrylamide and potassium persulphate were purchased from fluka, Sunflower stems were obtained from the research farm of Federal University of Technology, Akure (FUTA), Nigeria. The plant was identified and authenticated at the FUTA Herbarium, where a voucher specimen number FUTA0414 was assigned. Monochloroacetic acid and ethanol were obtained from Sigma Aldrich Chemicals. Ethanol, methanol and NaOH were used as purchased. All other reagents used were analytical grade and solutions were prepared with distilled water.

Sample Preparation

Preparation of powdered sunflower pith

The pith of the sunflower (SFP) was cleaned to remove debris, stones and other particles. The sample was then cut into smaller sizes, sun-dried and milled to fine powder and the final product was packed in different clean polythene bags and stored prior to use. The powdered sample was used for the compositional analysis of the lignocellulosic biomass.

Preparation of carboxymethyl sunflower pith (CMSFP)

The SFP was weighed and put in a 2000 mL beaker with 20% sodium hydroxide solution. The suspension heated and agitated at 70 ^{o}C for 5 h. The suspension was filtered using microcloth, and the obtained residue was washed with 90% ethanol, then transferred into a beaker with 15% sodium hydroxide solution in water bath and interfused by appropriate amount of monochloroacetic acid. The sample mixture was stirred and heated at 70 ^{o}C for 60 min, then filtered with 75% ethanol solution. The obtained chemically modified Sunflower pith was dried in an oven and stored in an air-container prior to use [9].

Preparation of CMSFP-g-PAm

CMSFP was dispersed in a 50 mL of distilled water. The suspension was then added into a 250 mL three-necked flask equipped with magnetic stirrer, reflux condenser, and nitrogen line in a water bath. A series of samples with different amounts of CMSFP, MBA, and KPS were prepared by the following steps: Nitrogen gas was bubbled into the solution for a few minutes. An appropriate amount of KPS was added with vigorous agitation and temperature was maintained at 70 ^{o}C. After about 20 min, appropriate amount of Am and MBA were successively added under vigorous stirring and nitrogen atmosphere. After reaction for about 50 min, the obtained gel (CMSFP-g-PAm) was washed with distilled water and 90% ethanol several times, cut into pieces and dried at 70 ºC. The dried polymer product was milled, screened and kept in a desiccator prior to use [10].

Swelling Measurement

Water absorbency measurement

The dry hydrogel powder was accurately measured and soaked in 500 mL tap water at room temperature for a day to reach swelling equilibrium. The unabsorbed water was removed from the swollen samples by filtering through a mesh screen. The weight of the swollen samples was measured and recorded. The water absorbency was calculated in triplicate (three replicate) as follows:

$$Q_{eq} = \frac{M_2-M_1}{M_1} \tag{1}$$

where Q_{eq} is the water absorbency per gram of dried sample (g/g). M_1 and M_2 are the weights of the dry and swollen samples respectively [11].

Water retention measurement

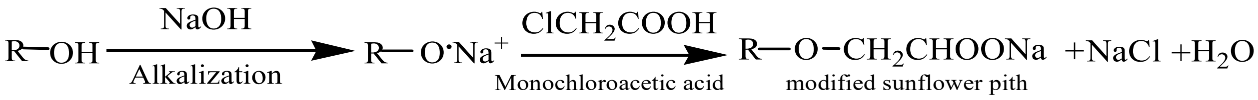
The synthesised hydrogel sample was immersed in tap water at room temperature to reach swelling equilibrium. Then, swollen gel was weighed (M_0) after the excess water was removed and reweighed at a regular time interval. The sample was weighed every 60 min for 8 h. The water retention capacity of the prepared hydrogel was determined according to Equation 2 [12].

$$R_{iT} = \frac{M_i}{M_0} x\ 100\% \tag{2}$$

Characterisation

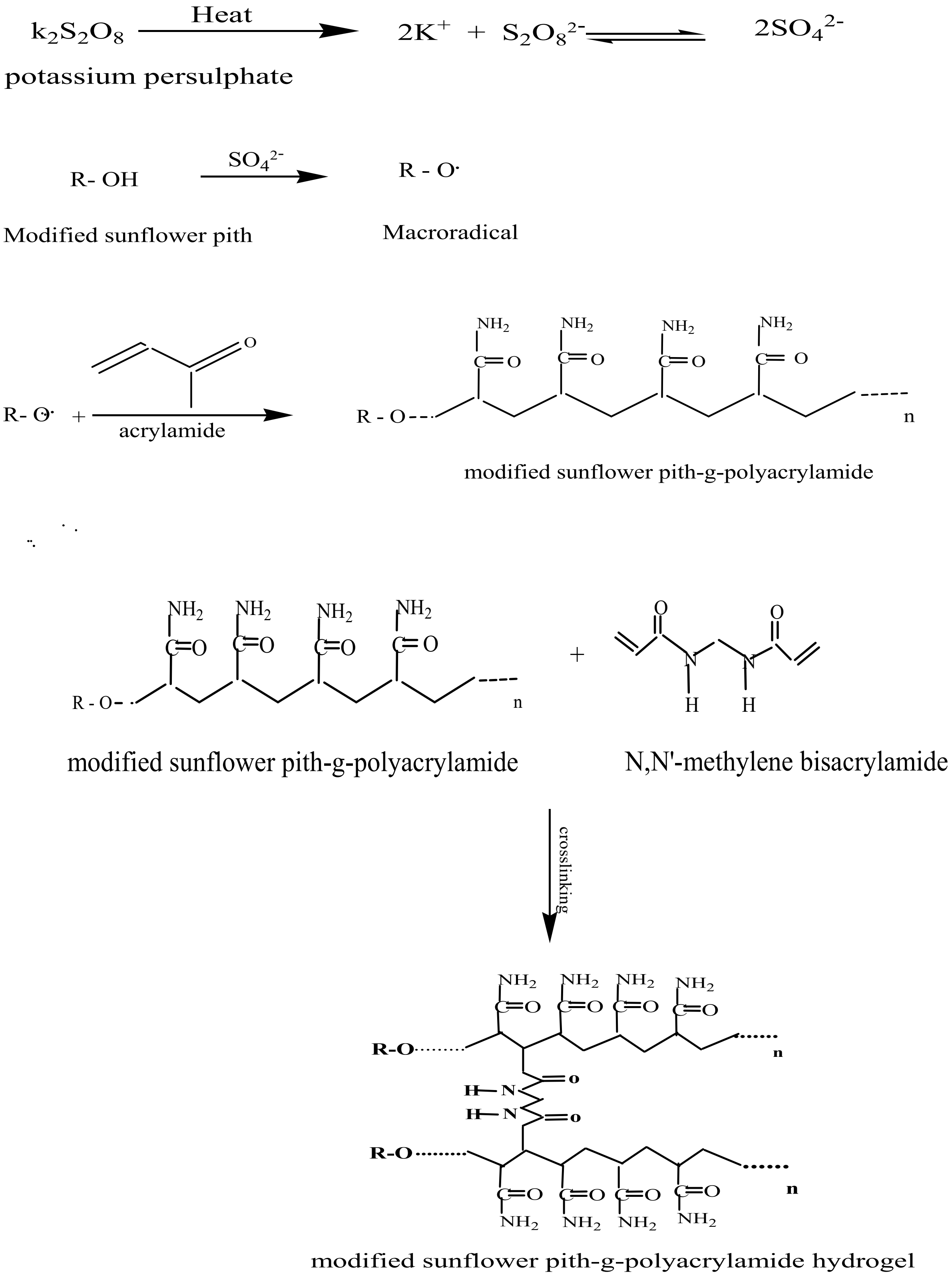
FTIR spectra were recorded on a FTIR Spectrometer in 4000 – 350 cm^{-1} region using KBr pellets. The surface morphologies and the structures of the prepared polymer gel were examined using scanning electron microscopy [13].

The proposed mechanism for the graft copolymerization of Am onto the CMSFP backbone in the presence of KPS and MBA is illustrated in Scheme 1.



$$R{-}OH \xrightarrow[\text{Alkalization}]{NaOH} R{-}O^{\cdot}Na^{+} \xrightarrow[\text{Monochloroacetic acid}]{ClCH_2COOH} \underset{\text{modified sunflower pith}}{R{-}O{-}CH_2CHOONa} + NaCl + H_2O$$

R = Sunflower pith (lignocellulosic biomass)



Scheme 1: Proposed reaction mechanism for the synthesis of CMSFP-g-PAm hydrogel

RESULTS AND DISCUSSION

Optimisation of the grafting variables

The various variables affecting the grafting conditions were optimised to obtain a hydrogel with maximum water absorbency. The images of *Tithonia diversifolia* pith and the synthesised hydrogel are shown in Plate 1.

Plate 1: (a) cut cross sections of *Tithonia diversifolia* pith and **(b)** Powdered *Tithonia diversifolia* pith **(c)** Image of the synthesised CMSFP-g-PAm hydrogel

Effect of biomass content

For the unmodified Sunflower pith-g-polyacrylamide and Carboxymethyl Sunflower pith-g-polyacrylamide hydrogels, Figure 1 shows that water absorbency initially increases with increasing SFP, and CMSFP contents, reaching an optimum, then declines.

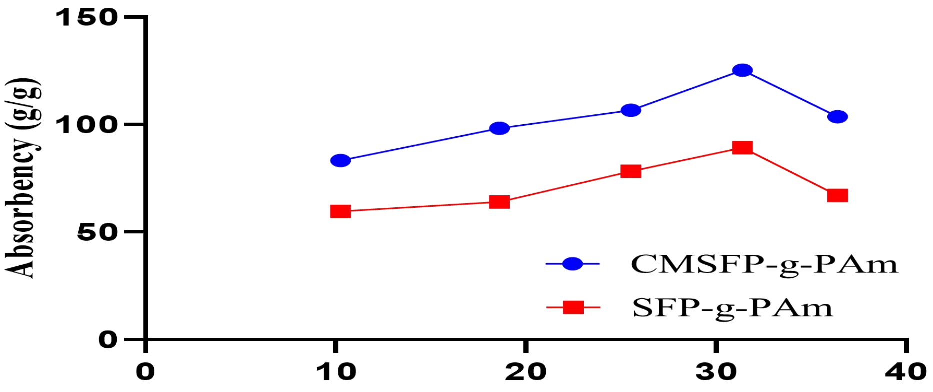


Figure 1: Effect of biomass content on water absorbency

This trend suggests an optimal biomass loading for maximum water uptake. Initially, as the biomass content increases from 10.26% to 31.4%, the availability of grafting sites on the SFP and CMSFP backbone likely increases, leading to a more robust and interconnected polymer network capable of imbibing more water. The peak absorbencies of 89.33 g/g and 125.33 g/g observed at 31.4% SFP and CMSFP respectively indicate an optimal balance between polymer grafting and hydrogel structure. However, exceeding this optimal loading (at 36.4%) leads to a significant decrease in absorbency. This decline can be explained by the potential for increased crosslinking density at higher concentrations. Excessive crosslinking reduces porosity of the hydrogel network, thereby limiting its ability to expand and absorb water [14]. The CMSFP-g-PAm hydrogel consistently demonstrates superior water absorbency compared to its unmodified SFP-g-PAm counterpart across all biomass content concentration. This substantial augmentation in water absorbency underscores the critical role of chemical modification which introduces anionic carboxyl groups (-COO-) onto the SFP backbone. These hydrophilic groups significantly enhance water absorption and position CMSFP-g-PAm hydrogels as promising candidates for various superabsorbent applications [15].

Effect of initiator content

The effect of amount of the initiator on the swellability of the hydrogel is shown in Figure 2.

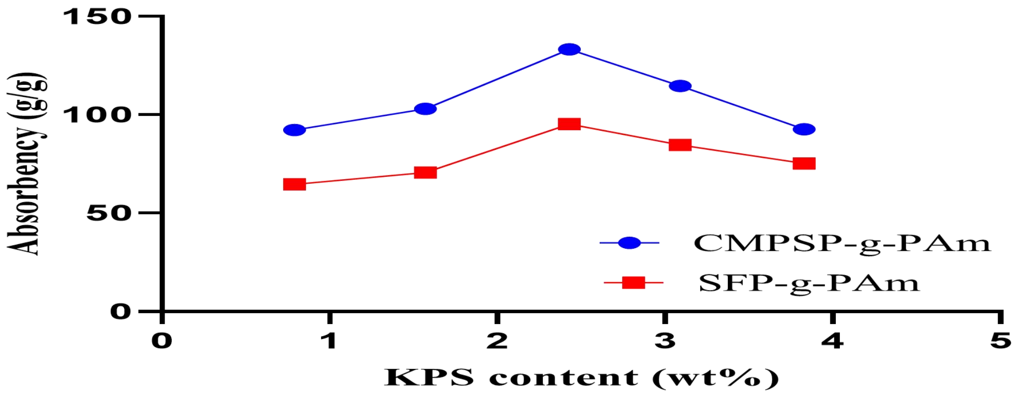


Figure 2: Effect of Initiator content on water absorbency

Generally, KPS acts as a source of free radicals, initiating the polymerisation of acrylamide (Am) onto the CMSFP and SFP backbone [16]. Water absorbency initially increases with increasing KPS content, reaching a maximum at 2.43% KPS, and then decreases with further increases in initiator concentration. Notably, the CMSFP-g-PAm hydrogel consistently demonstrated superior water absorbency compared to its unmodified counterpart across all KPS concentrations. At lower KPS concentrations (0.79% to 2.43% KPS), an increase in initiator content leads to the generation of a greater number of free radicals which initiate the grafting of Am monomers onto both the CMSFP and SFP backbones [17]. This results in a higher grafting efficiency, leading to a more developed polyacrylamide network. A robust and expansive polymer network can physically entrap and absorb more water due to increased polymer chain length and density of the absorbent network. The peak absorbency observed at 2.43% KPS for both hydrogels suggests that at this concentration, sufficient radicals are generated to form a well-crosslinked network with adequate porosity and appropriate chain length, maximising its water-holding capacity. However, beyond this optimal point, the water absorbency declines. This could be attributed to an excessively high crosslinking density [18].

Excessive crosslinking density causes reduction in the pore size within the hydrogel network, hindering the penetration and absorption of water molecules [15]. The consistently superior absorbency of CMSFP-g-PAm is primarily due to the presence of carboxymethyl groups (-COOH) introduced during the carboxymethylation process. These anionic groups, upon ionisation in water (forming -COO-), generate strong electrostatic repulsion within the polymer network, thereby enhancing the hydrogel's capacity to imbibe and retain substantial amounts of water [19].

Effect of cross-linking agent content

The cross-linking agent content, methylene bisacrylamide, plays a critical role in dictating the final swelling capacity and mechanical integrity of hydrogels. The effect of crosslinker content on the absorbency of SFP-g-PAm and the CMSFP-g-PAm is presented in Figure 3. From the results, water absorbency for both SFP-g-PAm and CMSFP-g-PAm initially increases as the MBA content rises from 0.078% to 0.16%. Both hydrogels exhibit their maximum water absorbency at an MBA concentration of 0.16 wt%. SFP-g-PAm reached 98.0 g/g, while CMSFP-g-PAm achieved an impressive 152.0 g/g. This suggests that a moderate level of crosslinking promotes higher water retention. At low crosslinker concentrations, the hydrogel network has larger pore sizes and increased flexibility, allowing for greater swelling and water uptake [18]. Insufficient crosslinking, however, can lead to a weak network structure that is prone to dissolution and premature degradation, limiting the overall water absorbency [16]. However, as the MBA concentration continues to increase beyond 0.16 wt%, a progressive reduction in swellability is observed in the synthesised graft copolymer. This decline can be attributed to the formation of a more tightly crosslinked network with smaller pore sizes and reduced chain flexibility [20]. Notably, the CMSFP-g-PAm hydrogel consistently demonstrates significantly higher water absorbency compared to SFP-g-PAm at every corresponding MBA concentration. This suggests that the carboxymethylation modification plays a crucial role in enhancing the hydrogel's water retention capacity.

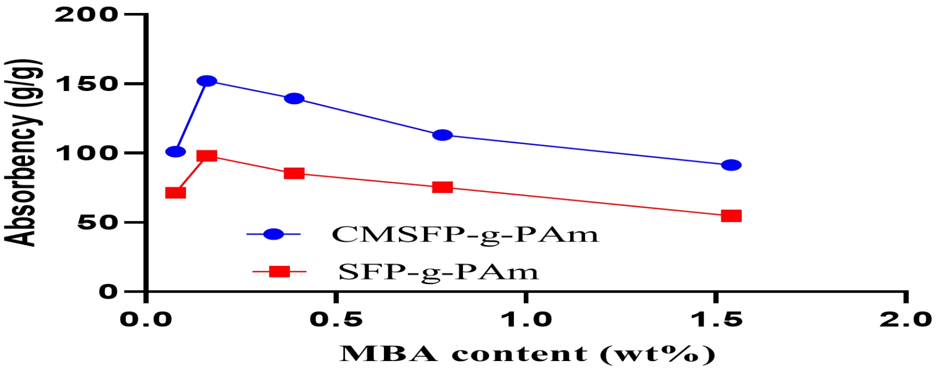


Figure 3: Effect of cross-linking content on water absorbency

Water retention capacity

Figure 4 shows the results of the study conducted to investigate the water retention capabilities of two synthesised hydrogels derived from sunflower pith: the unmodified Sunflower pith-g-polyacrylamide hydrogel and its carboxymethylated counterpart,

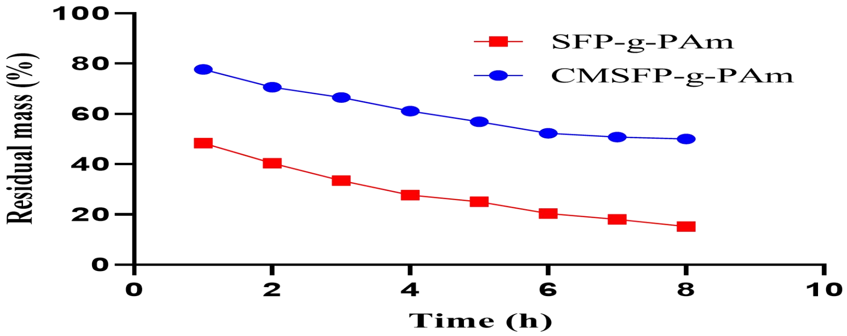


Figure 4: Water retention capacity of CMSFP-g-PAm and SFP-g-PAm

Carboxymethyl sunflower pith-g-polyacrylamide hydrogel. The results demonstrate a difference in their ability to retain absorbed water over an extended period of 8 h, with the CMSFP-g-PAm dramatically outperforming the SFP-g-PAm. The finding reveals a consistent decline in the percentage residual mass of water for both hydrogels over the 8 h period, a natural consequence of evaporation. However, the magnitude and rate of this decline, as well as the absolute residual water mass, differed significantly. The SFP-g-PAm hydrogel started with a residual water mass of 48.33% after 1 h. This relatively moderate initial retention suggests that while the polyacrylamide grafting enhances hydrophilicity and swelling compared to raw pith, its network primarily relies on physical entrapment and the inherent hydrophilic nature of polyacrylamide (PAm) [21].

Over time, its water retention capacity rapidly diminished and retained a mere 15.23% of its initial absorbed water after 8 h. The rapid decline indicates a weaker binding force between the water molecules and the polymer network. In contrast, the CMSFP-g-PAm hydrogel exhibited a significantly higher water retention capacity throughout the study. It began with 77.67% residual water mass after 1 h. This initial high retention immediately highlights the superior performance conferred by carboxymethylation. While it also lost water over time, the rate of loss was considerably slower, and the retained mass remained much higher, 50.03% after 8 h. The exceptional performance of CMSFP-g-PAm is directly attributable to the introduction of highly polar carboxymethyl groups onto the SFP backbone. They are capable of forming multiple strong hydrogen bonds with water molecules, significantly increasing the overall hydrophilicity of the polymer network [22].

FTIR analysis

The FTIR spectroscopy analysis provides crucial insights into the chemical structures of unmodified Sunflower pith-g-polyacrylamide and Carboxymethyl sunflower pith-g-polyacrylamide. The FTIR spectra of the synthesised hydrogels are illustrated in Figure 5. The broad and intense absorption bands at 3779.84 cm^{-1} and 3416.0 cm^{-1}, in FTIR spectrum of SFP-g-PAm, are primarily attributed to the O-H stretching vibrations of cellulose, hemicellulose, and lignin present in the sunflower pith, along with adsorbed moisture. It also encompasses the N-H stretching vibrations of the primary amide groups from the grafted PAm [23]. The band at 2926.42 cm^{-1} corresponds to the C-H stretching vibrations of both the aliphatic methyl and methylene groups [24]. The absorption peak at 1597.30 cm^{-1} represents the CN bending vibration (Amide I band) of the amide groups in PAm. The absorption band at 1380 cm^{-1} is typically assigned to C-H bending vibrations [24]. The FTIR spectrum of CMSFP-g-PAm exhibits distinct changes compared to SFP-g-PAm.

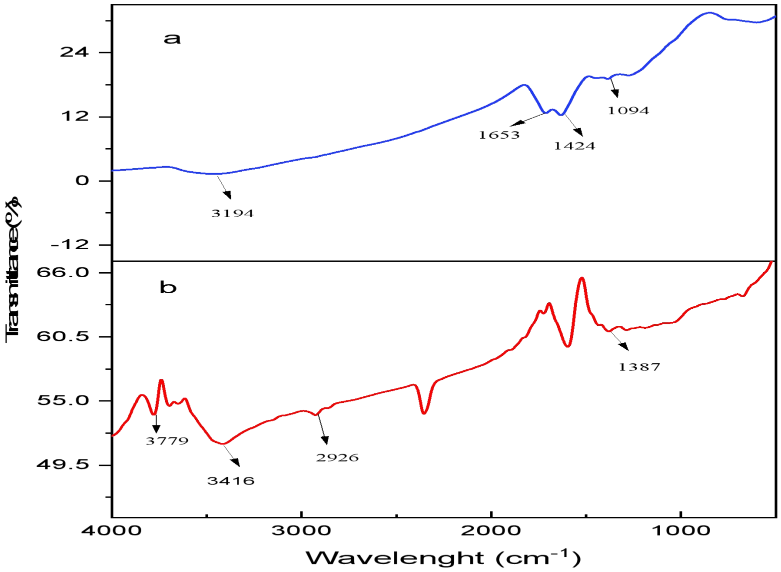


Figure 5: FTIR spectra of (**a**) SFP-g-PAm (**b**) CMSFP-g-PAm

The broad band at 3194.65 cm^{-1} is due to O-H stretching (from the hydroxyl groups on sunflower pith) and N-H stretching (from PAm). However, its shift to a lower wavenumber compared to SFP-g-PAm can indicate changes in the hydrogen bonding network due to the introduction of the carboxymethyl groups [25]. Similar to SFP-g-PAm, the band at 2924.9 cm^{-1} corresponds to C-H stretching vibrations [24]. The absorption peak at 1653.29 cm^{-1} is a critical peak for confirming carboxymethylation. It represents the asymmetric stretching vibration of the carboxylate anion ($–COO^{-}$). The band at 1424.92 cm^{-1} further supports the presence of the carboxylate group, corresponding to the symmetric stretching vibration of the carboxylate anion ($–COO^{-}$). The strong absorption band at 1094.41 cm^{-1} is characteristic of the C-O stretching vibration in the C-O-C glycosidic linkages [26]. In conclusion, the FTIR spectra provide clear spectroscopic fingerprints that validate both the carboxymethylation and the subsequent grafting of polyacrylamide onto sunflower pith, demonstrating the successful synthesis of the target modified biopolymers.

SEM analysis

The SEM images of the synthesised hydrogels are presented in Figure 6. Scanning electron microscopy analysis of unmodified Sunflower pith-g-polyacrylamide typically reveals a dense, less porous surface with relatively small and isolated pores. Conversely, the Carboxymethyl sunflower pith-g-polyacrylamide hydrogel image exhibits a significantly enhanced porous morphology, characterised by numerous, larger, and well-interconnected pores appearing as a sponge-like network. This marked increase in porosity for CMSFP-g-PAm is likely attributed to the hydrophilic carboxymethyl groups, which promote greater water absorption during synthesis, influencing the subsequent pore formation during drying. Such an extensive and interconnected porous structure in CMSFP-g-PAm is significant, as it facilitates improved swelling capacity, faster mass transfer and diffusion rates, and offers a larger surface area for adsorption or loading, enhancing the hydrogel's utility in various applications [27].

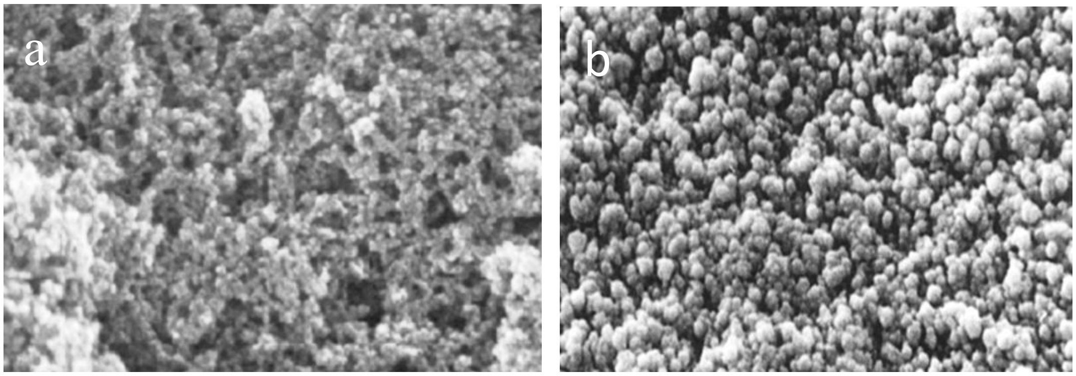


Figure 6: SEM images of **(a)** CMSFP-g-PAm **(b)** SFP-g-PAm

CONCLUSION

This study successfully demonstrated the viability of utilising pulverised native sunflower pith (SFP) for the synthesis of novel hydrogel materials through a free radical copolymerisation approach. A key aspect involved modifying the SFP through carboxymethylation, yielding carboxymethylated sunflower pith (CMSFP), which was subsequently copolymerised into hydrogels. The carboxymethylated sunflower pith-g-polyacrylamide (CMSFP-g-PAm) hydrogel exhibited superior water absorbency, achieving a maximum of 152 g/g. This represents a significant enhancement compared to 89.33 g/g for the unmodified SFP-g-PAm hydrogel, highlighting the positive impact of carboxymethylation on the material's hydrophilic properties. The optimum reaction conditions to obtain maximum water absorbency were found to be: MBA 0.16wt%, KPS 2.43wt%, and CMSFP/SFP 31.40wt%. Furthermore, the CMSFP-g-PAm hydrogel also demonstrated an improved capacity for water retention over an 8-hour period, characterised by a higher percentage of residual retained water. Characterisation *via* FTIR Spectroscopy confirmed the successful grafting and synthesis of the hydrogel structures. Complementary SEM analysis provided visual evidence of well-developed, interconnected porous networks within the hydrogels. This study clearly establishes carboxymethylated sunflower pith as a highly promising and sustainable precursor for the development of high-performance hydrogels. The significant improvements in water absorbency and retention, coupled with a desirable porous morphology, position these bio-based hydrogels as excellent candidates for various sustainable applications, particularly within agricultural systems for improving soil water management and nutrient delivery.

Acknowledgements

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