CELLULOSE CHEMISTRY AND TECHNOLOGY

PREPARATION OF A HYDROGEL COMPOSITE FROM RECYCLED CELLULOSE AND ITS USE FOR UREA RELEASE

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The aim of this study has been to develop a hydrogel composite based on recycled cellulose extracted from used baby diapers. The prepared composite was intended for agricultural applications, specifically for the controlled release of fertilizer (urea). To this end, used baby diapers were processed to extract cellulose, which was then sterilized and treated with acetic acid. The recycled cellulose was mixed with starch and citric acid, followed by precipitation with zinc ions to form the hydrogel composite. The resulting hydrogel composite had a heterogeneous structure due to the mixing of various components and exhibited a degree of thermal stability. The material demonstrated a very high water absorption capacity, achieving a swelling ratio of 260% within 4 hours, and released urea rapidly into the aqueous medium (40% after 2 hours). Urea release was governed by a simple diffusion mechanism. Growth tests on barley plants indicated that the urea-containing hydrogel promoted greater growth in both length and width, compared to the plants grown without the hydrogel, with more pronounced differences observed in length. The controlled release of urea from the hydrogel is crucial for optimizing barley plant growth. This study opens up promising avenues for waste recovery and the development of sustainable materials for agriculture.

Keywords: baby diapers, cellulose extraction, material composite, urea release, barley

INTRODUCTION

Cellulose is a biopolymer consisting of extensive chains of glucose units interconnected through β-1,4-glycosidic bonds. It is a fundamental component of plant cell walls and is recognized as the most prevalent polymer on Earth. Since its discovery in the 19th century, cellulose has garnered significant attention, leading to extensive research and a variety of applications in both industrial and medical fields.[1] The applications of cellulose span a wide spectrum, from basic uses to more complex ones. In the paper industry, cellulose serves as a critical raw material for the production of paper and cardboard. It also plays a vital role in the textile sector, particularly in cotton and linen, where it constitutes the primary component of these natural fibers.[2] Beyond its traditional roles, cellulose is being investigated as a potential source of biofuels due to its abundance and renewable characteristics. Through a process known as biomass conversion, cellulose can be transformed into ethanol and other biofuels.[3] In the medical domain, cellulose and its derivatives find utility in the pharmaceutical industry for the manufacture of capsules and tablets. For instance, derivatives like hydroxypropylmethylcellulose (HPMC) are employed as binders and thickeners in drug formulations. Additionally, cellulose fibers are utilized in producing medical materials, such as dressings and wound care products, which ensure fluid absorption and facilitate wound healing.[4,5,6]

Cellulose recycling is an important process that plays an important role in promoting environmental sustainability and reducing waste. Cellulose can be extracted from a variety of sources, including used paper, commercial paper products, agricultural waste, wood waste, natural fibers, industrial waste, food materials and biofuels.[7] The recycling process of waste paper, such as newspapers, magazines and writing paper, begins with the collection and sorting of these materials, and treatment is necessary to remove impurities, such as inks and adhesives.[8] The paper is also shredded, mixed with water to form pulp

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and may be reprocessed into new paper products, such as cardboard, tissue and printing paper.

By recycling waste paper, including various packaging, and food and beverage boxes, the need to cut down trees and consume natural resources is reduced.[9] Also, agricultural residues, such as crop straws and plant stalks, as well as fruit and vegetable skins, can also be used to extract pure cellulose, which can be then applied in papermaking and for various other applications.[11] Natural fabrics, like cotton and linen, are also a rich source of cellulose, thus, used clothes can be recycled to reduce fiber waste.[10] Extracting cellulose from such diverse sources can significantly reduce waste, while saving natural resources. In addition, such an approach creates employment opportunities in the collection, processing and manufacturing sectors, and encourages technological innovation to boost local economies, contributing to efficient and sustainable development.[12] By promoting recycling technologies and raising awareness about the importance of material reuse, we can reduce waste, conserve natural resources and achieve concrete environmental and economic benefits.[13]

This work focuses on the preparation and characterization of a new hydrogel composite developed by the cross-linking of cellulose extracted from used baby diapers and starch in the presence of zinc ions. This initiative has in mind the application of the hydrogel in agriculture, to improve soil fertility and boost crop productivity, through an approach that reduces waste and enhances environmental sustainability.

EXPERIMENTAL

Materials

In this study, cellulose fibers were extracted from used baby diapers, utilizing a range of chemical reagents. Urea (N_2H_4CO), with a molecular weight of 60 g/mol, was sourced from (Sigma Aldrich), in pellet form, with a purity of ≥99%. Sodium carbonate (Na_2CO_3), in the form of a white crystalline powder, with a purity of ≥99%, and zinc chloride ($ZnCl_2$), a white crystalline solid, with a molecular weight of 136.28 g/mol and a purity of ≥98%, were also purchased from Sigma Aldrich. Sodium hydroxide (NaOH), (Sigma Aldrich), was available as pellets and prepared as a 1 mol/L (1N) volumetric solution, with a purity of 98% and a molecular weight of 40.00 g/mol. Calcium chloride ($CaCl_2$), sourced from Labosi, was provided as a white crystalline solid with a purity of ≥99%. Citric acid ($C_6H_8O_7$) was obtained from Biochem Chemopharma, in the form of a white crystalline powder, with a purity of ≥99.5%. Lastly, hydrochloric acid (HCl) at a concentration of 37% was acquired from Analar Normapur, as a colorless liquid with a purity of ≥37%. Additionally, phosphate buffer solution (pH = 7.2) was obtained from Sigma Aldrich, with a purity of ≥99%. Starch, also sourced from Sigma Aldrich, was provided in powder form with a purity of ≥98%. These reagents played crucial roles in the extraction and characterization processes throughout the research.

The soil used in the seed germination study was obtained from the nearby area of the University of Chlef, Chlef, Algeria. The soil of the region (longitude 1.2891036 and latitude 36.1693515) is characterised as fertile, with a reddish hue and a pH of 6 to 7.

Cellulose extraction procedure

To extract cellulose, a soiled baby diaper weighing 10 g was initially autoclaved at 121 °C for 20 minutes. Following the autoclaving process, the superabsorbent polymer (polyacrylic acid) was removed from the diaper, and the remaining diaper fragments were ground using a blender. The fragments then underwent acid treatment in a 9% (v/v) acetic acid solution at 120 °C for 15 minutes. To facilitate the dissolution process, cellulose was isolated through vacuum filtration and subsequently purified by multiple washes with distilled water. This process yielded a white powder, with a total of 3.66 g of cellulose extracted from the original 10 g of diapers.[14]

Preparation of hydrogel composite

To prepare the hydrogel composite, 5 g of recycled cellulose was mixed with 3.5 g of sodium hydroxide in 200 mL of distilled water in a beaker. The mixture was heated to 70 °C, while being stirred magnetically for 30 minutes. Subsequently, 5 g of starch was incorporated into the mixture, followed by the addition of 4.5 g of citric acid to serve as a cross-linking agent. The pH of the solution was then adjusted to 7 using a sodium hydroxide solution. Afterward, 1 g of zinc chloride was introduced to the mixture at 70 °C and it was stirred for an additional 4 hours. The resulting precipitate was allowed to cool, thoroughly washed, and then dried under vacuum conditions.

Characterization

Infrared spectroscopy

The FTIR spectra of the prepared hydrogel, extracted cellulose and starch were recorded using an Alpha-Bruker instrument, by the KBr pellet technique. The measurements were made in the spectral range of 400 to 4000 cm^{-1} with a resolution of 1 cm^{-1}.[15]

Scanning electron microscopy (SEM)

The morphology of the composite surfaces was examined using a scanning electron microscope (Quanta 250 FEG), with an accelerating voltage of 3 kV and a spot size of 6 nm.[16]

Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was carried out to determine the thermal stability and degradation behaviour of the hydrogel composite. Samples were characterised using a TAG-Q500 thermogravimetric analyser. Approximately 6 mg of sample was placed in the instrument in a controlled environment. The samples were heated from 30 °C to 400 °C at a rate of 10 °C/min.[17]

Differential scanning calorimeter (DSC)

The thermal properties of the composite were assessed using a Shimadzu DSC-60 differential scanning calorimeter (DSC). Approximately 6.0 mg of composite was weighed, hermetically sealed and temperature equilibrated at room temperature for 1 hour. The system was calibrated using indium metal, with an empty aluminum dish serving as a reference. The analysis was carried out over a temperature range of 25-400 °C, with a heating rate of 10 °C/min.[18]

Swelling study

About 0.5 g of dried sample was weighed, and immersed in distilled water at 37 °C. After determined time intervals, the swollen sample was removed from the water, extra moisture from its surface was wiped off with filter paper and it was weighed again. The process was repeated 3 times.[19] The water uptake was calculated according to Equation (1):

$$S = \frac{M - M_j}{M_j} \times 100\% \qquad (1)$$

where M and M_j are the weight of swollen and dried samples, respectively.

Study of urea release in water

A sample of the hydrogel composite (0.5 g) was placed in contact with 100 mL of a urea solution (2.5 w/v) for 24 hours. Then, to assess the release of urea from the hydrogel composite, a 0.150 g sample was immersed in 100 mL of water. Constant, slow stirring was maintained using a magnetic stirrer. Aliquots of 0.5 mL were taken every hour and diluted to a final volume of 5 mL with phosphate buffer solution to determine the amount of substance released at each time interval by the spectrophotometric method.[20,21]

Release kinetics analysis

The Korsmeyer-Peppas equation (2) was used to analyze the mechanism of urea release from the hydrogels,[22] as a function of the value of the diffusion exponent (n).[23] This exponent is used to classify the release process into three categories: simple diffusion ($n < 0.45$), where urea is mainly released by diffusion through the hydrogel matrix; non-Fickian transport ($0.45 < n < 0.89$), which combines diffusion and relaxation of the hydrogel structure, making the process more complex; and dominant matrix relaxation ($n > 0.89$), where release is mainly controlled by relaxation of the hydrogel structure rather than by diffusion:[24]

$$\mathrm{Mt/M\infty} = K * t\text{^}n \qquad (2)$$

Effect of hydrogel composite on plant growth

To investigate the effects of the hydrogel on barley seed germination and growth, a controlled experiment was conducted with two sets of growth media: one containing a mixture of 0.5 g of urea-loaded hydrogel and soil in a ratio of 1:3 (hydrogel:soil), and the other containing only soil. Each cup was sown with 2-3 barley seeds placed approximately 1-2.5 cm deep, followed by a thin layer of soil to cover the seeds.

Before planting, the hydrogel was conditioned and mixed with the soil to create a moisture-retaining growth medium, which is critical for seedling establishment.[25,26] The cups were equipped with drainage holes to prevent waterlogging and were irrigated twice daily using a sprinkler mechanism to maintain optimal soil moisture, while minimizing disturbance to the seeds. The plastic cups were placed in a warm, well-lit environment protected from direct sunlight to create favorable conditions for germination.

RESULTS AND DISCUSSION

Infrared spectroscopy

The FTIR spectra of the hydrogel composite, starch, and cellulose are shown in Figure 1. In the spectrum of starch, there is broad band around 3300 cm^{-1}, which is attributed to the stretching vibration of the O-H bond.[27] The peak around 2900 cm^{-1} belongs to the stretching vibration of C-H bonds, which are prevalent in the glucose ring structure. Also, the band corresponding to C=O stretching is observed at 1625 cm^{-1}. The band at 1450 cm^{-1} represents CH_2 and that at 1048 cm^{-1} is assigned to C–O–C stretching.[28]

The infrared spectrum of cellulose exhibits several characteristic absorption bands. The intense and broad peak around 3400 cm^{-1} is assigned to the stretching vibrations of the abundant hydroxyl (O-H). The peaks observed in the 2900 cm^{-1} region correspond to the symmetric and asymmetric stretching vibrations of the methylene (CH_2). The band at 1650 cm^{-1} can be attributed to the bending vibrations of the hydroxyl (O-H) groups, particularly those associated with absorbed water molecules within the cellulose structure. The peaks around 1425 cm^{-1} and 1375 cm^{-1} are associated with the bending vibrations of the methylene (CH_2) and methine (CH) groups respectively. The strong band at 1060 cm^{-1} is assigned to the stretching vibrations of the C-O-C.[29]

In the spectrum of the prepared hydrogel, the band at 3328 cm^{-1} and 3253 cm^{-1} was found to be associated with the stretching vibrations of -OH and -CH groups, respectively.[30] Examination of the -CH, COOH and O-H regions of the infrared spectrum provides valuable structural information relating to the structure of the hydrogel. The bands at 3375-3340 cm^{-1} correspond to intramolecular hydrogen bonds OH---O.[31]

In addition, the peak shift from 3391 cm^{-1} (cellulose/starch) to 3343 cm^{-1} (cellulose-zinc/starch-zinc)[32] indicates attachment of Zn^{2+} ions to OH hydroxyl groups, creating new OH---Zn^{2+} interactions.[33] The incorporation of citric acid between the cellulose and starch chains tends to shift the band located at 3340 cm^{-1} towards wave numbers around 3419 cm^{-1}.[34]

The absorption of the -OH group arising from the absorbed moisture in the extracted cellulose was found at 1673 cm^{-1}.[35] Furthermore, the study showed the presence of CH_2 bending vibrations in cellulose and starch at 1591 cm^{-1},[36] O-H bending vibrations at 1451 cm^{-1},[37] and pyranose ring C-O-C skeletal vibrations at 1151 cm^{-1}.

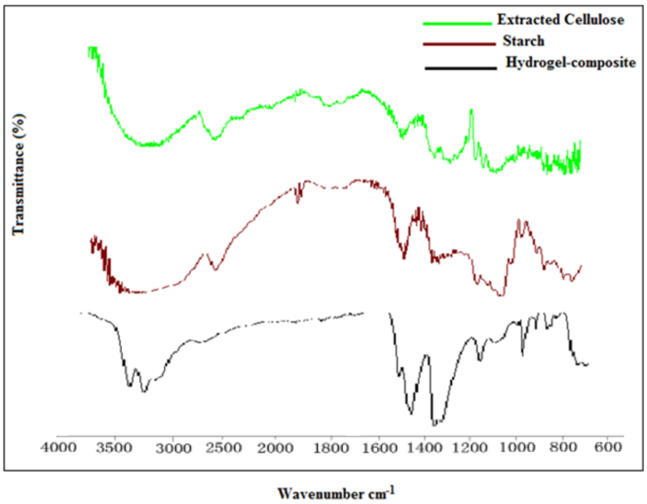


Figure 1: Infrared spectra of the prepared hydrogel composite, extracted cellulose and starch

Thermogravimetric analysis

The thermal stability of the composite was examined by thermogravimetric analysis at a temperature rise rate of 10 °C/min. The percentage mass loss of the samples was plotted as a function of temperature, from 30 °C to 400 °C. The thermograms of the samples are shown in Figure 2. This analysis enables the thermal degradation points to be determined and the resistance of the materials to high temperatures to be assessed, providing crucial information about their thermal behaviour.[38] Although stable at moderate temperatures, cellulose decomposes significantly from 200 °C onwards. This degradation accelerates sharply above 300 °C, leading to almost total loss of mass at 400 °C.

The thermogram of the hydrogel clearly shows that degradation takes place in two stages. The first weight loss occurs between 30 and 145 °C, mainly due to the removal of moisture and adsorbed water from the composite. The percentage weight loss in this first stage is estimated to be between 8 and 10%. In the second stage, significant weight loss occurs between 145 and 400 °C. This degradation is mainly due to oxidative degradation of the cellulose and starch chains, leading to breakage of the chain fragments and monomers present in the two macromolecules. In all cases, the percentage weight loss is around 53%, indicating that half of the degradation occurs when the samples are heated in this temperature range. The addition of zinc to the composite leads to improved thermal properties; these results are in good accordance with other findings in the specialized literature.[39]

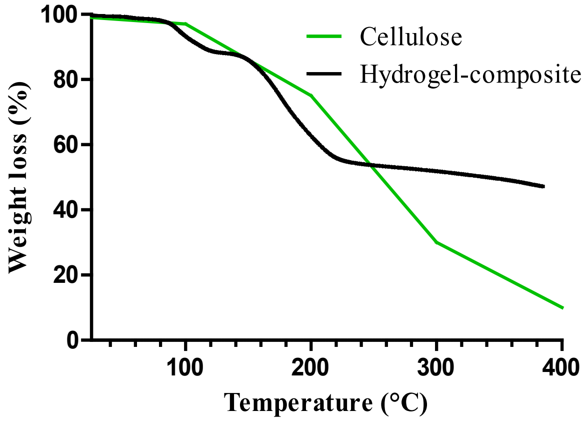


Figure 2: TGA curves of the hydrogel composite and cellulose

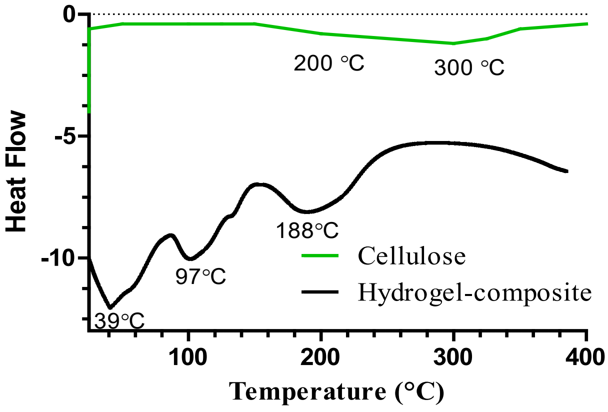


Figure 3: DSC curves of the hydrogel composite and cellulose

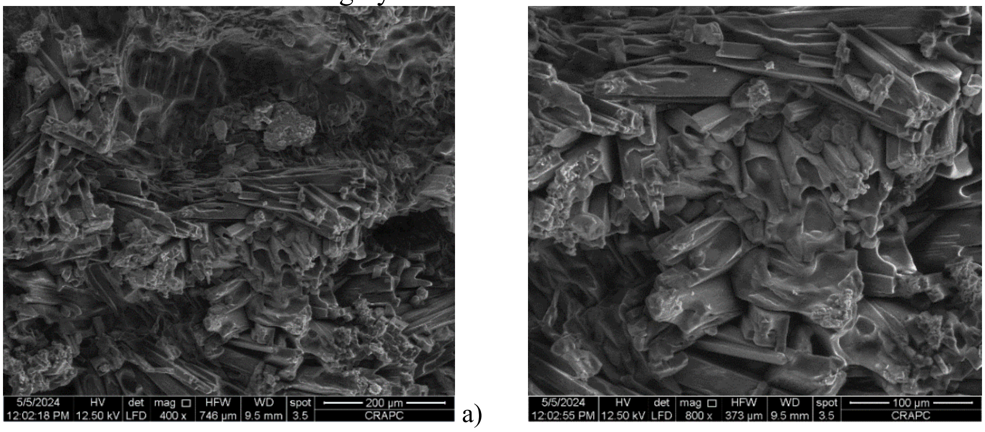
Differential scanning calorimetry (DSC)

The DSC curve (Fig. 3) of the hydrogel composite shows three key peaks: the initial endothermic peak around 39-80 °C, indicating moisture loss and structural changes, while the second, more pronounced peak marks the onset of gelatinization around 97 °C of the starch.[40] Additional endothermic events around 188 °C reflect the onset of the polymeric structure decomposition, providing information on the stability and behavior of the composite under heat, which is crucial for applications in materials science and biochemistry.[41] Also, the endothermic peak at around 200 °C indicates heat-induced depolymerization of the cellulose.

The addition of zinc salts significantly alters the thermal properties of cellulose-based materials, as revealed by differential scanning calorimetry (DSC) analysis.[42] Zinc chloride is an effective swelling agent for cellulose, modifying its thermal behavior and structural stability. Composites prepared with zinc ions are thermally stable.[43] In particular, the addition of zinc ions to highly basic mixtures considerably increases the specific surface area of cellulose, thereby improving its thermal properties.[44] In cellulose-zinc complexes, melting temperatures shift. This change in thermal transitions, such as the endothermic peaks, is associated with melting and dehydration.[45]

Scanning electron microscopy

The scanning electron microscopy (SEM) images of the composite, illustrated in Figure 4 (a to d), indicate a heterogeneous structure. Notably, images in Figure 4(c) and 4(d) display tetragonal crystal fibers characterized by a smooth and uniform surface, along with porosity. Furthermore, the starch and cellulose appear to be uniformly distributed throughout the matrix, implying effective compatibility among the various phases. This finding suggests that a controlled crystallization process has taken place, promoting the development of organized structures.[46]



a)

b)

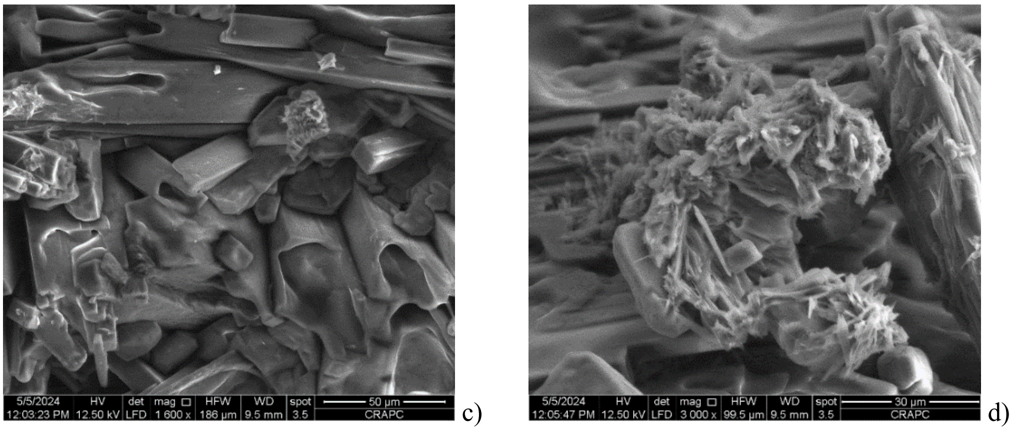


Figure 4: SEM micrographs of hydrogel at different magnification

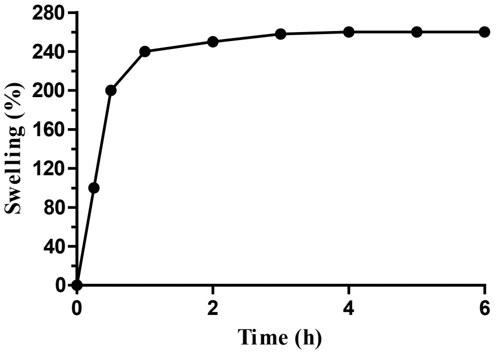


Figure 5: Swelling kinetics of the hydrogel

Swelling kinetics

Our experiment extended our understanding of the swelling behavior of a polymeric material prepared in distilled water. The data provided in Figure 5 show a rapid increase in the percentage swelling of the hydrogel over the first few hours, followed by stabilization. These results indicate that this material reached its swelling equilibrium after 4 hours, with a water absorption of 260%, compared with its original size. This level of absorption can be attributed, in part, to the structure of the starch and cellulose components. The presence of hydroxyl groups in the starch structure facilitates water absorption via hydrogen bonds. This swelling capacity has implications, particularly in agricultural fields, where these materials can be used to improve water retention and reduce irrigation needs.

Urea release into water

The graph in Figure 6, showing the release of urea from the composite over time, reveals a profile that is typical of many controlled-release systems. At the start of the experiment, there is a rapid initial phase (2 h), marked by a rapid increase in the percentage of urea release in the medium with a value of 40%. This phenomenon can be explained by the release of urea from the surface of the material, which diffuses rapidly into the environment, as well as by the presence of large pores within the material, facilitating this diffusion.[47] After this initial phase, the rate of urea release decreases considerably, leading to a stabilization of the release percentage (after 48 hours). This transition to a slow release phase can be attributed to several factors: firstly, the decrease in the concentration gradient between the inside of the material and the outside environment, which slows down the diffusion process.[48,49] Secondly, if the urea is bound to specific sites within the material, the gradual exhaustion of these sites may also limit the rate of release. Finally, over time, the structure of the material may evolve, reducing the size of the pores and thus limiting urea diffusion.

The Korsmeyer-Peppas kinetic model has been used to study the release of urea from the hydrogel into water and soil. The kinetic release parameters are listed in Table 1 and represented graphically in Figure 7.

The experimental results show that Fick's diffusion model is the most suitable for describing the mechanism of controlled release of urea from the hydrogels in water. Indeed, the coefficient of determination (R^2) greater than 0.85 and the value of the diffusion exponent (n) less than 0.45 indicate that the release of this fertilizer is governed by a simple diffusion mechanism, in accordance with Fick's law of diffusion. Urea is gradually released from the hydrogel as it moves through its pores and channels, driven by the concentration gradient between the inside and outside of the matrix. In addition, the swelling of the hydrogel can influence the rate of release by modifying the size of the pores and the diffusion distance.

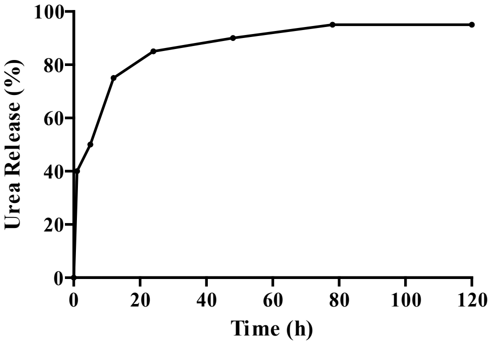
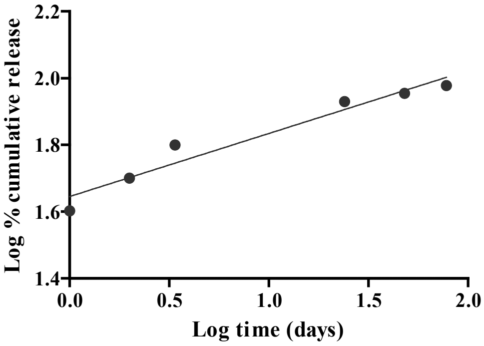


Figure 6: Urea release kinetics into water



Figures 7: Korsmeyer-Peppas model of urea release

Table 1
Parameters of the Korsmeyer-Peppas model for urea release in water

Kinetics model	Parameters
Korsmeyer–Peppas	R^2 = 0.9601 n = 0.214 k = 1.769

Effect of hydrogel on the growth of barley plants

There is growing interest in the use of hydrogel composites in agriculture due to their ability to improve the water retention capacity of the soil and provide favorable environment for growth. This study aims to assess the impact of the developed hydrogel composite on the growth of barley plants. Throughout the experiment, soil moisture levels were regularly monitored to assess the effect of the hydrogel on the germination success and plant growth, and the results indicated differences in plant growth metrics – with regard to height and width – between the two growth conditions.

The data shows that the urea-containing hydrogel has a significant impact on barley plant growth, both in length and width.[50,51] Initially, on day 5, the plants growing in regular soil (without hydrogel) were 5 cm long, while those in the hydrogel supplemented soil were only 2.5 cm long. However, as time passed, the hydrogel began to release urea, allowing the plants to catch up. Between days 5 and 8, the length of the plants became closer, reaching 6 cm for the plants with the hydrogel and 8 cm for those without. On day 10, the plants with hydrogel reached 8 cm, almost identical to the 8.1 cm of the plants without hydrogel. From day 11, the plants with hydrogel began to outgrow those without, reaching 9 cm compared with 8.7 cm, thanks to the nitrogen released by the urea. Finally, on day 13, the plants with hydrogel measured 9.8 cm, while those without reached 8.9 cm, demonstrating that the hydrogel favors optimal release of urea over a prolonged period, which is essential for superior growth.

In terms of width, on day 5, the plants growing without hydrogel were 0.4 cm wide, compared with 0.3 cm for those with hydrogel. Between days 5 and 7, the width of the plants with the hydrogel increased to 0.5 cm, while those without hydrogel increased to 0.45 cm. On day 8, both sets of plants reached an identical width of 0.5 cm, indicating that the urea was sufficiently available to allow equivalent growth. Between days 8 and 11, the width of the plants with the hydrogel increased slightly to 0.57 cm, while those without reached 0.52 cm. On day 13, both sets of plants had a final width of 0.57 cm for those growing in the hydrogel supplemented soil and 0.52 cm for those in regular soil, showing that the hydrogel contributed to a slight increase in plant width from day 11.

These results suggest that the urea-containing hydrogel promotes the growth of barley plants in length and width, although the differences in width are less marked than those observed for length. The controlled and prolonged release of nutrient (urea) from the hydrogel is crucial for optimizing barley plant growth, resulting in a greater height compared with plants grown without the hydrogel.[52,53]

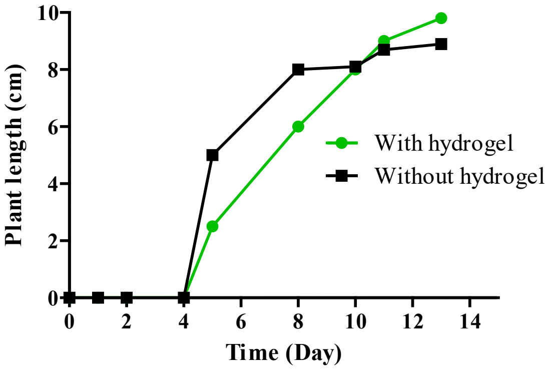


Figure 8: Evolution of length of barley plants growing in hydrogel supplemented soil and regular soil

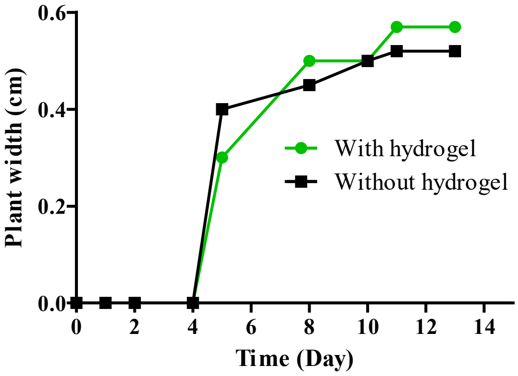


Figure 9: Evolution of width of barley plants growing in hydrogel supplemented soil and regular soil

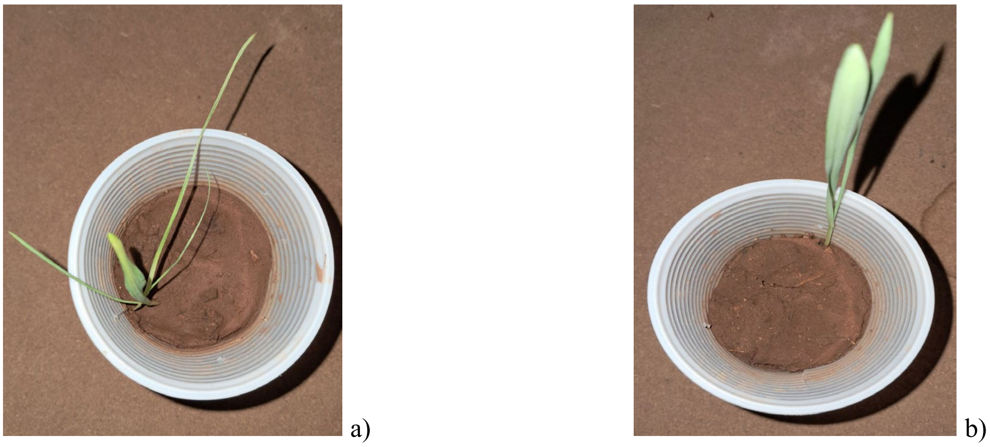

a) b)

Figure 10: Barley plants growing in regular soil and hydrogel supplemented soil

CONCLUSION

This work demonstrated the possibility of recycling cellulose from baby diapers to create a high value-added hydrogel composite for agricultural purposes. The recycling process included sterilization and acid treatment to purify the cellulose, which was then combined with starch, citric acid and zinc ions. Physico-chemical characterisation confirmed the formation of a complex polymer network, while swelling studies showed typical hydrogel behaviour, favouring hydration. In addition, the release of urea was governed by a simple diffusion mechanism, consistent with Fick's model, which is promising for its use as a controlled release system for fertilisers. In addition, the effect of the hydrogel on barley growth was studied and the results showed that the urea-containing hydrogel stimulates plant growth in both length and width. However, the differences observed in width were less pronounced than those in length. The controlled and prolonged release of nutrients, in particular urea, from the hydrogel is essential for optimising the growth of barley plants, resulting in greater plant height than in the case of plants grown in regular soil (without hydrogel) under the same conditions.

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