CHARACTERIZATION OF ENVIRONMENTALLY FRIENDLY CTS/PVA HYDROGEL APPLIED AS A GROWING MEDIUM FOR PLANTS

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ABSTRACT

Hydrogel is a hydrophilic material capable of retaining large amounts of water. It has recently been considered an attractive material for modern agricultural development, particularly as a growing medium for plants. This article takes advantage of the abundant chitosan (CTS) source in Vietnam to combine with polyvinyl alcohol (PVA) to make a hydrogel that can carry nutrients to plants without soil. Hydrogels were synthesized by a freeze-thaw method based on the ratio of CTS and PVA. In addition, the properties of hydrogels, such as mechanical strength, swelling degree, gel fraction, and rheology, were determined. SEM and FTIR were used to analyze the chemical structure and morphology of the hydrogel. Moreover, the samples were also tested for germination and growing plants. The results show that the CTS/PVA (2-3) hydrogel shows many advantageous features, including a swelling index of 483 %, ultimate tensile strength of 0.30 MPa, and stability under 65 °C. This study can expand the potential of hydrogels in sustainable agricultural development.

Keywords: Hydrogel, polyvinyl alcohol, chitosan, growing medium.

1. INTRODUCTION

Hydrogels are three-dimensional, hydrophilic polymer networks capable of absorbing and retaining large amounts of water relative to their mass. Thanks to this property, hydrogels exhibit various applications in agriculture, food, the biomedical field, cosmetics, drug delivery, engineering, and construction [1-4]. Recently, in the agricultural field, research on hydrogel has increased greatly [5-8]. Hydrogels gained recognition primarily due to their capacity as controlled carriers for pesticides, fertilizers, and water [9]. In this regard, hydrogels derived from environmentally friendly and biodegradable polymers are receiving much attention because they possess the capacity to transform agricultural methodologies significantly without affecting the environment [1].

Chitosan is a natural polymer that has non-toxic, biocompatible, and biodegradable characteristics. Chitosan is a polycationic polymer synthesized by deacetylation of chitin, commonly collected from crustaceans' exoskeletons, particularly shrimps and crabs [10-12]. CTS-based hydrogels exhibit diverse applications in the field of agriculture, such as antimicrobial properties in plants, increasing plant growth, and allowing controlled release of agrochemicals [1]. In contrast to chitosan, PVA is a synthetic polymer. However, it has been noted as a hydrophilic, biocompatible, low cytotoxic, degradable polymer [13-15]. Many studies were performed on PVA in hydrogel form, all of which strongly substantiated its suitability for applications in agriculture and the food industry [9].

In the context of taking advantage of low-cost chitosan, which is created from the abundant source of seafood waste in Vietnam, and the demand for sustainable agriculture regarding a new approach as a hydrogel, CTS and PVA were objects that the article wanted to exploit to promote their potential. However, the disadvantage of each chitosan or PVA as a hydrogel is its weak mechanical strength [13,16]. Importantly, hydrogels composed of multiple polymers can have mechanical strengths greater than that of a single polymer because of the increase in the number of crosslinking points within the network. This is a result of robust intermolecular interactions from different polymer chains [17]. Therefore, combining chitosan and PVA is based on intermolecular hydrogen bonds of the functional groups, such as -OH and $-NH_2$, in their structures that can improve mechanical strength [1].

There are many methods of making hydrogels. Based on the cross-linking method, hydrogels can be classified into chemical hydrogels and physical hydrogels. Physical hydrogels have a transient junction comprising physical interactions, such as ionic interactions, hydrogen bonding, and crystallization. Compared with chemical hydrogels, physical hydrogels often have lower mechanical strength, but they are receiving much attention due to their high purity, facile gelation process under mild conditions, and the absence of external cross-linking agents [2, 13]. This article aims to study one of the physical crossing-linking methods. It is the repeated freeze-thaw (F/T) cycle method. Briefly, this technique produces stable, physically cross-linked hydrogels by inducing polymers' self-assembly (mainly linking of hydrogen) via the presence of crystalline regions during freezing, which concentrates polymer chains. Meanwhile, the thawing process creates persistent crystalline junctions. It can be said that the F/T method is simple, environmentally friendly, and easily scalable [18-21]. Therefore, the application of this method to synthesize CTS/PVA hydrogel can enhance its features and reduce production costs [22].

This paper aims to study the effects of CTS and PVA components on the properties of CTS/PVA hydrogels. Based on analytical methods such as SEM, FTIR, tensile strength, rheology, and water content, the features of the hydrogels were evaluated, especially mechanical strength and swelling capacity. In addition, germination and growth of Brassica chinensis var. parachinensis plant (Yu Choy plant) tests on hydrogels were performed to examine their suitability as plant media.

2. MATERIALS AND METHODS

2.1. Materials

Poly(vinyl alcohol) (PVA) (98% hydrolyzed, the average degree of polymerization is 1700-1800) was obtained from Oxford (India). Chitosan (CTS) (medium molecular weight) originated in Vietnam. All the chemicals were of analytical reagent grade without further purification.

2.2. Methods

2.2.1. Preparation of CTS/PVA hydrogel

PVA was first dissolved in deionized water (10% w/v) at 85 °C. CTS 3 wt.% was dissolved in 2 wt.% aqueous acetic acid at room temperature. Next, all the solutions were stirred to make homogeneous solutions. Then, two solutions were mixed with volume ratios (CTS/PVA) of 1:1, 1:2, and 1:3 by high-speed stirring for 2 hours. Finally, the CTS/PVA solution was poured into a mold and subjected to 3 cycles of freezing at -15 °C for 24 hours and thawing at room temperature for 8 hours.

2.2.2. Mechanical properties

The mechanical properties of hydrogels were tested using a Zwick Roell Z010 (Germany) machine. The gel samples (evaporated moisture to 50% of their original mass) were cut into a rectangular shape, and the tensile strength analysis was performed at a stretching rate of 10 mm/min with an initial load of 5g.

2.2.3. Wetting, swelling behavior, and gel fraction

Water content (WC) could be expressed by the following equation (1) [23]:

$$WC\ (\%) = \frac{W_w - W_d}{W_d} x100\% \qquad [1]$$

where W_w and W_d correspond to the weight of the as-prepared and dry samples, respectively.

The swelling properties of the hydrogels were measured by following equation (2), as in the previous document [24]:

$$SI\ (\%) = \frac{W_s - W_d}{W_d} x100\% \qquad [2]$$

where W_d and W_s are the weights of dried and swollen samples, respectively.

Gel fraction (GF) represents the amount of this crosslinked network within the hydrogel. It is typically determined by the following equation (3) [24, 25]. The pieces of the hydrogel samples were dried for 12 hours at 60 °C in an oven (W_o). Then, they were soaked in distilled water till a constant weight. The hydrogels were dried again at 60 °C to reach the weight (W_e).

$$GF\ (\%) = \frac{W_o - W_e}{W_o} x100\% \qquad [3]$$

2.2.4. Characterization

Structures of the polymer were analyzed by Fourier-transform infrared spectroscopy (FTIR) on a Frontier of Perkin Elmer spectrophotometer. The surface morphologies of hydrogels were investigated using an emission scanning electron microscope (SEM, JSM-6510LV). The viscosities and rheological data were obtained from TA Instruments (USA). The value of viscosity was measured at 25 °C. The parameters for the rheological test were a cone plate sensor system performed at an angular frequency of 10 rad/s.

2.2.5. Germination and Growth Experiment

The germination and root development of the Yu Choy plant were tested on hydrogels impregnated with nutrients (N, P, K). First, the mass of fertilizer (N, P, K) is weighed with the same content and put into the same volume of water to soak the gel. The gel absorbed the nutrient water and swelled. Then, the seeds were sown on the hydrogels, and the shape and roots of the plants were assessed over time.

3. RESULTS AND DISCUSSION

Three samples with different ratios of CTS/PVA (v/v) were investigated, which are CTS-PVA (1-3), CTS-PVA (2-3), and CTS-PVA (3-3), implying the volume fraction of CTS to PVA is 0.33, 0.66, and 1.0, respectively. This is attributed to the strong hydrogen bonds formed between the large number of hydroxyl and amino groups of CTS and the hydroxyl groups of PVA.

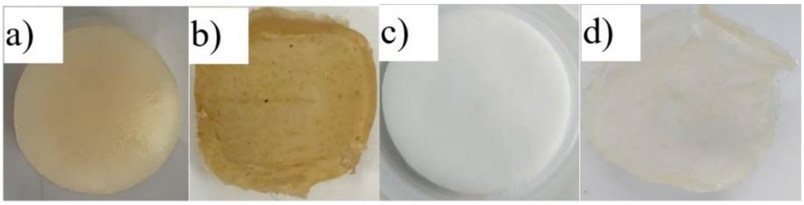

Figure 1. Appearances of CTS/PVA (2-3) and PVA hydrogel: a,c) gel as-prepared, and b,d) gel after drying

In observation, the obtained CTS/PVA and PVA samples had uniform shapes. They contained large amounts of water and did not completely collapse after drying (Fig. 1 b,d). Among the pure polymer samples, only the PVA was able to form a gel (Fig.1c), whereas the CTS failed to do so. This shows that the combination of CTS and PVA is reasonable.

3.1. Characterization of the hydrogels

Fig. 2 shows the viscosity of the CTS, PVA, and CTS/PVA polymer solution. Specifically, the blending of CTS with PVA had a higher viscosity than the pure CTS (0.30 Pa.s) and PVA (0.69 Pa.s), except the sample CTS/PVA (2-3) had a value of 0.61 Pa.s (within the range of CTS and PVA). It can be said that there is an interaction between CTS and PVA, which changes the viscosity value. However, the binding between the two substances does not depend on the CTS content but may depend on the orderly arrangement between the two polymer chains. That may be the reason why the CTS/PVA (2-3) sample has low viscosity.

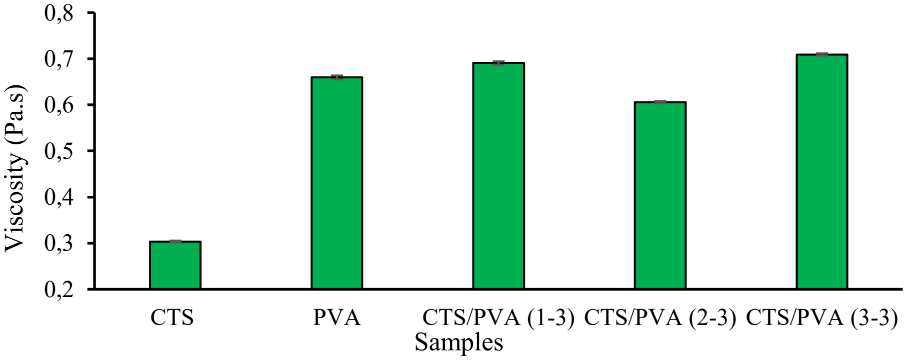

Figure 2. Viscosity of CTS, PVA, and CTS/PVA polymer solutions

According to the previous study [26], the FTIR data of the CTS and PVA showed the representative peaks, including the broadband (3300–3600 cm^{-1}) corresponding to –OH groups, the absorption peaks of –C=O group (amide type 1) at 1650 cm^{-1} and the –NH group (amide type 2) at 1590 cm^{-1} for chitosan and the weak peaks at 1699 cm^{-1} are associated with stretching vibrations of –C=O bonds of PVA. Whereas, the CTS/PVA showed the interaction between the functional groups of the two polymers in Fig.3. The –C=O and N–H absorption peaks of the CTS/PVA sample are very similar to the CTS infrared spectra, with the intensity being lower at wavelengths of 1638 and 1619 cm^{-1}, respectively. In addition, the strong band of the –OH group placed at the wavenumber center (3445 cm^{-1}) shifted between the pure wavenumbers of PVA (3603 cm^{-1}) and CTS (3300 cm^{-1}).

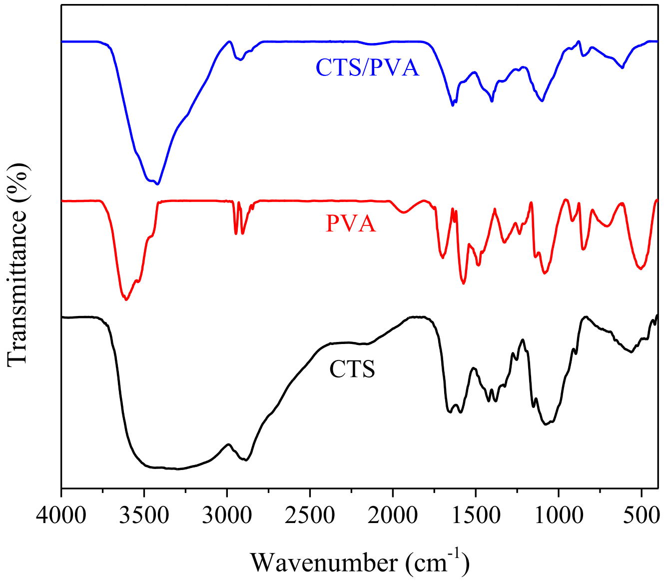

Figure 3. FTIR data of CTS, PVA, and CTS/PVA

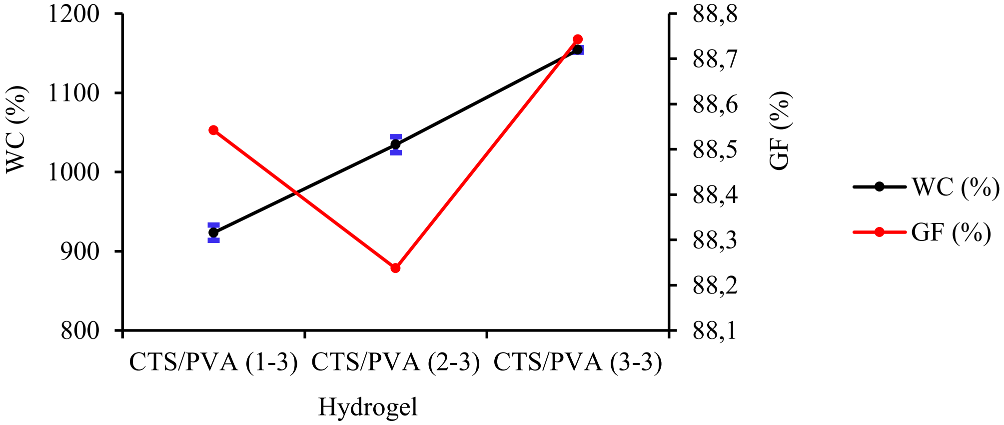

Figure 4. Water content and gel fraction of CS/PVA hydrogels

After implementing three cycles of F/T, the formed gel samples were examined for water content, swelling degree, and gel fraction. The water content in Fig. 4 increased with the CTS ratio, and all three samples contained a high initial water content of 923%, 1034%, and 1154%, respectively. Consistent with the viscosity results, the CTS/PVA (2-3) sample had the lowest gel content (88.2%) and the highest swelling index (483.6%) in Figs. 4,5. It can be explained that the number of bonds between the two polymers (CTS, PVA) was not as high as in the other samples.

As is known, the gel fraction of a hydrogel refers to the degree of crosslinking within the hydrogel structure. A higher gel fraction generally signifies a more robust hydrogel with a denser network of crosslinks [25]. So, owing to the highest gel content of 88.7%, the CTS/PVA (3-3) sample could have many bonds between the two polymers. It can be explained that the higher the chitosan content, the greater the number of functional groups, so more hydrogen-bonding interactions with PVA functional groups. However, the water content and swelling degree of the CTS/PVA (3-3) sample are also high, accounting for 1154% and 370.6%, respectively. The hydrogel swelling is directly related to porosity and pore size. This behavior is attributed to increasing the crystallinity in gels, suppressing the swelling ratio [22]. It is predicted that the CTS/PVA(3-3) sample formed a large pore in the structure cause of no crystalline zones, so the swelling degree and water content were high. Therefore, this sample may not achieve good mechanical strength. Meanwhile, the CTS/PVA (1-3) sample had the lowest chitosan content among the three samples; the gel percentage (88.5%) was high, and the swelling index (264.4%) and water content (923%) were the lowest. It suggests strong interactions between the polymers, creating a crystallinity region and high mechanical strength.

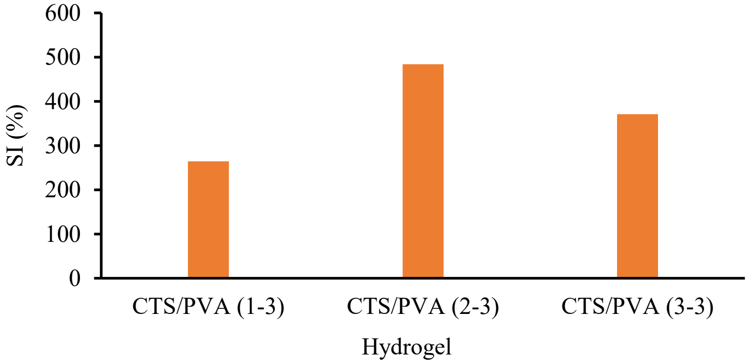

Figure 5. Swelling index of CS/PVA hydrogels

3.2. Mechanical properties of CTS/PVA gels

The tensile stress-strain curves of CTS/PVA hydrogels are illustrated in Fig. 6. Tensile stress varied nonlinearly with strain and showed typical viscoelastic behavior. The tensile strength depended on CTS content; the more CTS, the lower the mechanical strength. As can be seen, the CTS/PVA (3-3) gel has a poor value, with the lowest ultimate tensile strength (UTS) of 0.18 MPa. In contrast, the CTS/PVA (1-3) gel shows the highest UTS (1.13 MPa) and the elongation at break (223%) results. The sample of CTS/PVA (2-3) obtained an average of UTS (0.30 MPa). The mechanical strength results are in complete agreement with the hydrogel properties data analyzed above. This shows that a large amount of CTS leads to poor polymer chain arrangement, which can reduce mechanical strength.

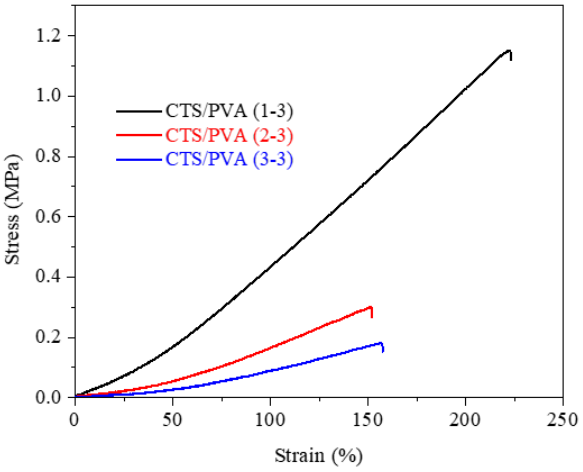

Figure 6. Stress-strain curves of the hydrogels at room temperature

Based on the application orientation as a plant growth environment and green agricultural development, the CTS/PVA (2-3) sample was selected for the next experimental research due to its good mechanical strength and swelling. This selection is based on the priority of swelling to be able to absorb large nutrients and water, ensuring plant growth while taking advantage of the abundant amount of CTS. Another reason for this selection is related to the control of nutrient release. According to the literature [10], hydrophilic polymers, such as polyvinyl alcohol, release active chemical compounds through diffusion, whereas chitosan releases active components by degradation and diffusion. Hoping that hydrogel CTS/PVA (2-3) will be a potential material for this application.

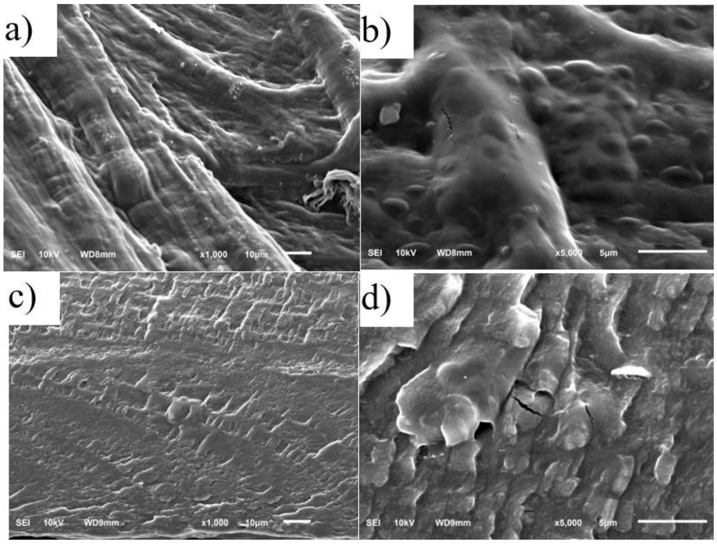

Figure 7. SEM images of cross sections of PVA in low magnification (a), PVA in high magnification (b), CTS/PVA (2-3) in low magnification (c), CTS/PVA (2-3) in high magnification (d)

Fig.7 displays the morphology of CTS/PVA (2-3) hydrogels with a relatively flat surface, a few-layered structure distributed uniformly in micro-pores. Whereas, the PVA images exhibit fiber bundles, and smooth appearance. This is another proof that the combination of CTS and PVA had changed the structure, created many pores, and increased the swelling degree.

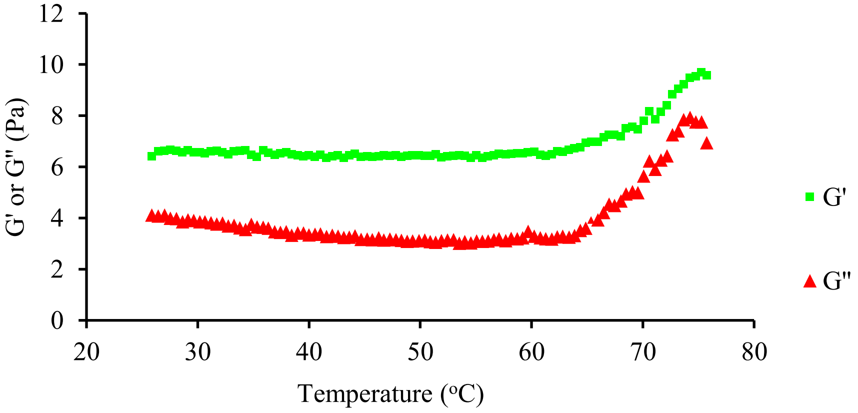

Figure 8. Storage G′ and loss G″ modulus of CTS/PVA (2-3) hydrogels in temperature

Furthermore, Fig. 8 shows the representative results regarding the viscoelastic characteristic of CTS/PVA (2-3). The results show that the elastic nature of the CTS/PVA, due to the storage modulus (G') was much higher than the loss modulus (G") over the temperature range. Under 65 °C, both G" and G" varied little, indicating a stable network structure for the hydrogel. However, from 75 °C, G" suddenly increases more quickly than that of G', implying that the hydrogel increases its viscosity behavior. It can be said that in Vietnam's climate conditions, the application of this hydrogel can be completely assured of the gel's durability. The result in Fig. 9 of the CTS/PVA (2-3) sample soaked in water for 1 month is also proof that the gel is stable. The surface still had a uniformly distributed structure, but there was looseness between the layers.

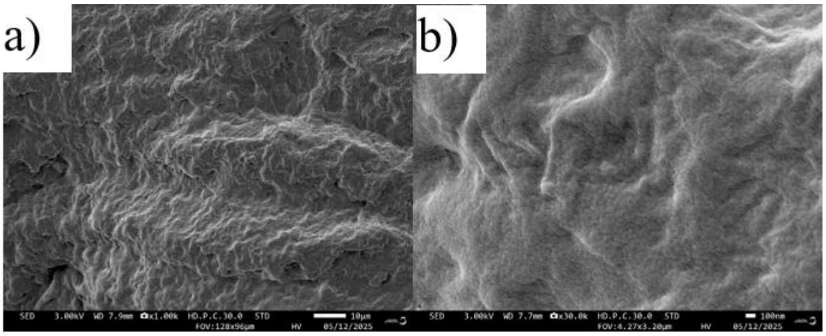

Figure 9. SEM images of CTS/PVA (2-3) immersing in water for 1 month: a) in low magnification, and b) in high magnification

3.3. Germination and Growth Experiment

In this study, the CTS/PVA (2-3) hydrogels were used agriculturally to determine the effect on the germination of Yu Choy seeds and the growth of roots of plants. All seeds are planted at random in the piece of hydrogel that absorbs nutrients (N, P, K). The results in Fig. 10 (a,b,c) show that the seeds germinated naturally and developed normally. After 12 days, the roots grew strongly, penetrated, and adhered well to the gel layer. The plants did not fall when the gel was turned upside down in Fig. 10d. In addition, it also showed that the mechanical strength of the gel was not significantly affected. Specifically, the gel did not tear or crack. This opens the strong potential for this hydrogel to develop in green agriculture.

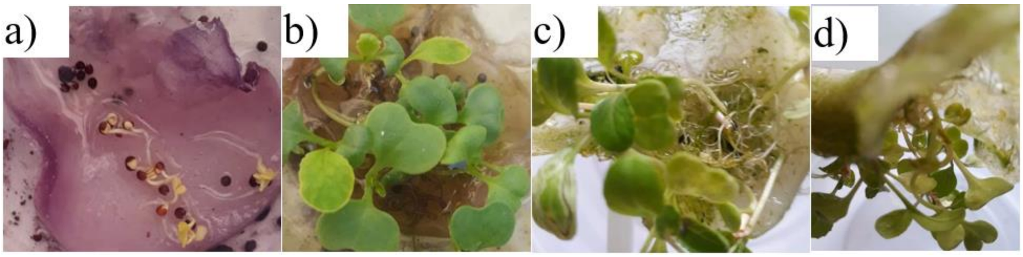

Figure 10. The photos of the seed germination and growing of Yu Choy plants in the CTS/PVA hydrogel after: a) 5 days, b,c) 12 days, and d) upside down the plants after 12 days

4. CONCLUSION

The combination of chitosan with PVA by the F/T method has successfully formed a hydrogel with high mechanical properties, opposite to the CTS. These hydrogels possessed a high swelling index, average from 260% to 480%, and high mechanical strength from

0.18 MPa to 1.13 MPa. The chitosan content did not mainly impact these two parameters, but the arrangement of the polymer chain interaction might reasonably create a stable hydrogel with good swelling. Through SEM images and rheological analysis, CTS/PVA (2-3) gel had good stability and a uniform surface with small pores arranged on the layer. Initial tests on the ability to grow Yu Choy seed on the gel showed normal germination, strong root generation, and penetration of the roots into the gel. Hopefully, with this property, CTS/PVA hydrogel can be further studied for its ability to absorb and release nutrients in a controlled manner for green agricultural development.

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TÓM TẮT

ĐẶC TÍNH CỦA HYDROGEL CTS/PVA THÂN THIỆN VỚI MÔI TRƯỜNG ĐƯỢC ỨNG DỤNG LÀM MÔI TRƯỜNG PHÁT TRIỂN CÂY TRỒNG

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Hydrogel là vật liệu ưa nước có khả năng hấp thu lượng lớn nước. Gần đây, hydrogel được quan tâm trong ứng dụng trong nông nghiệp hiện đại, đặc biệt là làm môi trường phát triển cây trồng. Bài viết này tận dụng nguồn chitosan (CTS) dồi dào tại Việt Nam để kết hợp với polyvinyl alcohol (PVA) tạo ra hydrogel vận chuyển chất dinh dưỡng đến cây trồng mà không cần đất. Hydrogel được tổng hợp bằng phương pháp đông lạnh-tan băng dựa trên tỷ lệ CTS và PVA. Bên cạnh đó, các tính chất của hydrogel như độ bền cơ học, mức độ trương nở, thành phần gel và đặc tính lưu biến được xác định. SEM và FTIR được sử dụng để phân tích cấu trúc hóa học và hình thái của hydrogel. Ngoài ra, các mẫu hydrogel cũng cho kết quả khả quan về thử nghiệm nảy mầm và phát triển cây cải. Kết quả cho thấy hydrogel CTS/PVA (2-3) thể hiện nhiều đặc điểm nổi bật, bao gồm chỉ số trương nở là 483%, độ bền kéo cực đại là 0,30 MPa và ổn định dưới 65 °C. Nghiên cứu này có thể mở rộng tiềm năng ứng dụng hydrogel trong phát triển nông nghiệp bền vững.

Từ khóa: Hydrogel, polyvinyl alcohol, chitosan, môi trường phát triển cây trồng.