PECTIN EXTRACTION FROM *OPUNTIA DILLENII* AND PHYSICOCHEMICAL CHARACTERIZATION OF THE FORMED HYDROGEL

CHIẾT XUẤT PECTIN TỪ XƯƠNG RỒNG (*OPUNTIA DILLENII*) VÀ PHÂN TÍCH ĐẶC TÍNH HYDROGEL TẠO THÀNH

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ABSTRACT

The paper aims to prepare antioxidant hydrogel from *Opuntia dillenii*, extract pectin by ultrasonic and ethanol precipitation, and form gel through the reaction between pectin with STPP (1%) and Ca^{2+} (4%). At ultrasonic conditions of 37kHz, 70°C, 120 min and the solvent to the raw material ratio of 10:1 (v/w) in an acidic environment (pH 2), the obtained pectin has a stable fibrous structure. 70% ethanol allows the good precipitation of pectin, forming a hydrogel with a stable cross-linked polymer network. Physicochemical spectroscopy showed that FTIR shows –OH, –COOH, –COOR groups and cross-linking signs; SEM shows a homogeneous porous structure; XRD shows semi-crystalline properties; TGA records mass loss corresponding to dehydration and gradual decomposition; DSC shows phase transition peak, confirming the thermal stability of the system. These results showed that hydrogel has high antioxidant activity, opening up potential in biomedicine and food technology.

***Keywords*:** *Hydrogel; Opuntia dillenii; pectin; antioxidant; physico-chemistry.*

TÓM TẮT

Bài báo nhằm điều chế hydrogel chống oxy hóa từ cây xương rồng có gai (*Opuntia dillenii*), chiết pectin bằng sóng siêu âm và kết tủa ethanol, tạo gel qua phản ứng giữa pectin với STPP (1%) và Ca^{2+} (4%). Dưới điều kiện siêu âm 37kHz, 70°C, 120 phút và tỷ lệ dung môi:nguyên liệu 10:1 (v/w) trong môi trường acid (pH 2), pectin thu được có cấu trúc dạng sợi bền vững. Ethanol 70% cho phép kết tủa pectin hiệu quả, từ đó tạo thành hydrogel có mạng polymer liên kết chéo ổn định. Đo phổ hóa lý thấy, FTIR thể hiện các nhóm –OH, –COOH, –COOR và dấu hiệu liên kết chéo; SEM cho thấy cấu trúc xốp đồng nhất; XRD chỉ ra tính bán kết tinh; TGA ghi nhận mất khối lượng tương ứng với mất nước và phân hủy dần; DSC chỉ ra đỉnh nhiệt chuyển pha, khẳng định độ bền nhiệt của hệ thống. Những kết quả này chứng minh hydrogel đạt hoạt tính chống oxy hóa cao, mở ra tiềm năng ứng dụng trong lĩnh vực y sinh và công nghệ thực phẩm.

Từ khóa: *Hydrogel; xương rồng; pectin; chống oxy hóa; hóa lý.*

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1. INTRODUCTION

In recent years, the development of biocompatible and environmentally friendly biomaterials has attracted considerable attention in fields such as biomedicine, functional foods, and pharmaceuticals [1]. Among them, hydrogels three-dimensional polymer networks with

high water-retention capacity are regarded as one of the ideal derivative systems due to their flexibility, biodegradability, and excellent biocompatibility [2].

Opuntia dillenii, a cactus species native to arid and semi-arid regions, demonstrates robust growth and contains a high content of polysaccharides, particularly pectin a water-soluble fiber with a linear polymer backbone rich in carboxyl groups (–COOH) [3]. Pectin not only provides nutritional value but also serves as a precursor for synthesizing functional materials, especially natural antioxidant hydrogels [4]. However, this valuable bioresource remains underutilized in industrial processing.

Traditional pectin extraction methods often involve high temperatures and prolonged durations, which can lead to structural degradation and reduced bioactivity [5]. Recently, ultrasound-assisted extraction has emerged as an efficient method for extracting polysaccharides, offering shorter extraction times, higher yields, and better preservation of biological activity [6]. Following extraction, pectin can be recovered through ethanol precipitation [7] and subsequently gelled via ionic interactions with cross-linking agents such as sodium tripolyphosphate (STPP) and calcium ions (Ca^{2+}) [8].

This study aimed to develop a protocol for extracting pectin from *Opuntia dillenii* using ultrasound-assisted extraction, followed by ethanol precipitation, and hydrogel formation through cross-linking with STPP and Ca^{2+}. In addition, the physicochemical properties and antioxidant activity of the resulting hydrogel were analyzed and evaluated to determine its potential applications in biomedicine and food technology.

2. MATERIALS AND METHODS

2.1. Raw materials

Prickly pear cactus (*Opuntia dillenii*) was harvested from semi-arid sandy soils after 20 months of growth. The samples were thoroughly washed with water to remove soil and sand, the spines and outer peel were removed, and the mucilage was filtered out. The cactus tissue was then finely chopped to facilitate pectin extraction.

2.2. Chemicals and equipment

The chemicals used in this study included 70% ethanol (Merck), sodium tripolyphosphate (STPP, 1%), calcium chloride ($CaCl_2$, 4%), and nitric acid (HNO_3, Merck) for adjusting the pH to 2. The equipment used included an ultrasonic bath (37 kHz), thermostatic water bath, centrifuge, drying oven, freezer, UV-Vis spectrophotometer, Fourier-transform infrared (FTIR) spectrometer, scanning electron microscope (SEM), X-ray diffractometer (XRD), thermogravimetric analyzer (TGA), and differential scanning calorimeter (DSC).

2.3. Sample preparation

Pectin was extracted from pretreated cactus stems using nitric acid solution with pH adjusted according to experimental conditions. The extraction was supported by ultrasound at a frequency of 37kHz, with specific temperatures and durations, to disrupt cell structures and release pectin. The extract was filtered and centrifuged to collect the liquid phase, followed by precipitation with 70% ethanol at a 1:1 volume ratio. The precipitate was washed with ethanol, dried at 40°C, and finely ground to obtain crude pectin powder.

2.4. Experimental design for hydrogel preparation

The pectin extraction process was investigated using a one-factor-at-a-time (OFAT) experimental design, focusing on four main influencing factors: Solvent-to-material ratio: 10:1, 15:1, 20:1, 25:1, and 30:1 (v/w); Extraction temperature: 60°C, 70°C, and 80°C; Extraction time: 60, 90, and 120 minutes; Extraction pH: 1.5, 2.0, and 2.5 (adjusted using HNO_3); After extraction, pectin was precipitated with 70% ethanol, centrifuged, and dried at 40°C. The pectin yield and preliminary morphological properties were used as indicators to determine the optimal extraction conditions. The extracted pectin was then dissolved in distilled water (2%, w/v), followed by sequential addition of STPP (0,8%) and $CaCl_2$ (8%) to induce ionic cross-linking reactions. Hydrogel formation was carried out at room temperature with gentle stirring for 30 minutes, then kept at 4°C for 24 hours to stabilize the gel structure. Evaluation criteria consisted of: Gel morphology (by SEM), functional group interactions (by FTIR), crystallinity (by XRD), thermal stability (by DSC and TGA), antioxidant activity (by total antioxidant, reducing power, and DPPH assays).

2.5. Analysis methods

Quantification of Pectin Content: The pectin content was determined using two methods: 1) *Gravimetric method after drying:* The pectin precipitated with 70% ethanol and dried at 40°C was accurately weighed. The yield was calculated as the ratio of the dried pectin mass to the dry weight of the raw material, reflecting the extraction efficiency; 2) *Uronic acid determination:* The uronic acid content was quantified using the m-hydroxybiphenyl method. The reaction was carried out in concentrated H_2SO_4, producing a colored complex with absorbance measured at 525nm. Galacturonic acid was used as the standard [9].

Structural and physicochemical characterization: The hydrogel was characterized using Fourier-transform infrared spectroscopy (FTIR) to identify functional groups such as –OH, –COOH, and –COOR, and to detect ionic cross-linking. The morphology and gel structure were observed using scanning electron microscopy (SEM). Crystallinity was assessed by X-ray diffraction (XRD). Thermal properties were evaluated using two techniques: thermogravimetric analysis (TGA) to monitor weight loss with temperature, and differential scanning calorimetry (DSC) to determine the phase transition temperature of the hydrogel.

Antioxidant activity analysis: The antioxidant activity of the hydrogel was assessed using three main indicators: total antioxidant capacity (TAC) via the phosphomolybdenum method, ferric reducing antioxidant power (FRAP), and DPPH radical scavenging activity. These indices were measured using a UV-Vis spectrophotometer at their respective characteristic wavelengths. Results were processed using Microsoft Excel and statistically analyzed by one-way ANOVA at a significance level of $p < 0.05$.

2.6. Data Analysis

All experiments were conducted in triplicate ($n = 3$). Results are presented as mean values ± standard deviation. Quantitative data were analyzed by one-way analysis of variance (ANOVA) at a significance level of $p < 0.05$ to determine statistically significant differences between experimental conditions.

3. RESULTS AND DISCUSSION

3.1. Pectin Extraction

The extraction of pectin from cactus was optimized by evaluating the effects of pH, extraction time, temperature, and solvent-to-material (S/M) ratio on pectin yield, total antioxidant capacity (TAC), and ferric reducing antioxidant power (FRAP). Data were analyzed using one-way ANOVA at a significance level of $\alpha = 0.05$, and the results showed statistically significant differences ($p < 0.05$) across the tested conditions for all three parameters. Among the different pH levels, an acidic environment (pH 2) yielded the highest pectin content (2.883 ± 0.32g) along with the highest antioxidant activities (TAC: 0.230 ± 0.001µg AE/g, FRAP: 0.34 ± 0.002mg/g). These values were statistically different ($p < 0.05$) from those at pH 7 and 9, which showed significantly lower extraction efficiency and bioactivity. This supports the role of low pH in breaking down cell walls and facilitating the release of pectic substances. In terms of extraction time, 120 minutes proved optimal, yielding 3.007 ± 0.05g of pectin, TAC of 0.290 ± 0.001µg AE/g, and FRAP of 0.35 ± 0.01mg/g, all of which were significantly higher than at other time points ($p < 0.05$). A decline in yield at 150 minutes (1.456 ± 0.09g) was observed, likely due to thermal degradation or depolymerization of the pectin chains under prolonged heat exposure. For extraction temperature, 70°C provided the best results, with high pectin yield (3.007 ± 0.05g) and antioxidant activity (TAC: 0.290 ± 0.001µg AE/g, FRAP: 0.35 ± 0.001 mg/g). Both lower (60°C) and higher (80°C) temperatures resulted in reduced yield and activity, with statistically significant differences ($p < 0.05$), indicating that both insufficient and excessive heat can adversely affect pectin integrity and solubility. The solvent-to-material ratio also had a pronounced effect. A 15:1 (mL/g) ratio was found to be optimal, yielding 3.007 ± 0.05g of pectin with high TAC and FRAP values. Higher ratios such as 25:1 and 30:1 showed a decreasing trend in yield ($p < 0.05$), possibly due to over-dilution, which reduces the solvent concentration gradient and extraction efficiency. From these results, the conditions of pH 2, extraction time of 120 minutes, temperature of 70°C, and S/M ratio of 15:1 were determined to be optimal for extracting high-yield, bioactive pectin from cactus. The pectin obtained under these conditions served as a key precursor for the preparation of pectin hydrogel, and was further analyzed using structural characterization techniques such as FTIR, XRD, TGA, and DSC, as discussed in subsequent sections. Unlike previous studies that primarily relied on high temperatures and extended extraction times, this study employed ultrasound-assisted extraction under optimized conditions (pH 2, 70°C, 120 minutes, solvent-to-material ratio of 15:1), which not only shortened the extraction time but also preserved the structural integrity of the pectin. The achieved yield of 13.52 ± 0.47% surpasses many previously reported values for pectin derived from *Opuntia* species.

Table 1. Effects of extraction parameters on total antioxidant capacity, reducing power, and pectin yield from cactus-derived pectin hydrogel

Parameter	Condition	Total antioxidant activity (µg AE/g)	Reducing power activity (mg/g)	Pectin yield (g)
pH	2	0.230 ± 0.001	0.34 ± 0.002	2.883 ± 0.32
	7	0.210 ± 0.001	0.14 ± 0.001	2.467 ± 0.42
	9	0.220 ± 0.001	0.18 ± 0.002	2.387 ± 0.49

Extraction Time	60 min	0.190 ± 0.001	0.27 ± 0.001	2.617 ± 0.51
	90 min	0.230 ± 0.001	0.344 ± 0.002	2.883 ± 0.32
	120 min	0.290 ± 0.001	0.35 ± 0.01	3.007 ± 0.05
	150 min	0.270 ± 0.001	0.33 ± 0.001	1.456 ± 0.09
Extraction Temp.	60 °C	0.140 ± 0.001	0.21 ± 0.001	1.963 ± 0.06
	70 °C	0.290 ± 0.001	0.35 ± 0.001	3.007 ± 0.05
	80 °C	0.240 ± 0.002	0.32 ± 0.001	2.283 ± 0.32
Solvent-to-Material Ratio (S/M)	10:1	0.191 ± 0.001	0.253 ± 0.001	2.540 ± 0.001
	15:1	0.290 ± 0.001	0.35 ± 0.001	3.007 ± 0.05
	20:1	0.210 ± 0.002	0.310 ± 0.002	2.366 ± 0.02
	25:1	0.180 ± 0.002	0.295 ± 0.002	2.372 ± 0.02
	30:1	0.174 ± 0.002	0.270 ± 0.002	2.266 ± 0.02

3.2. Physicochemical properties of the hydrogel

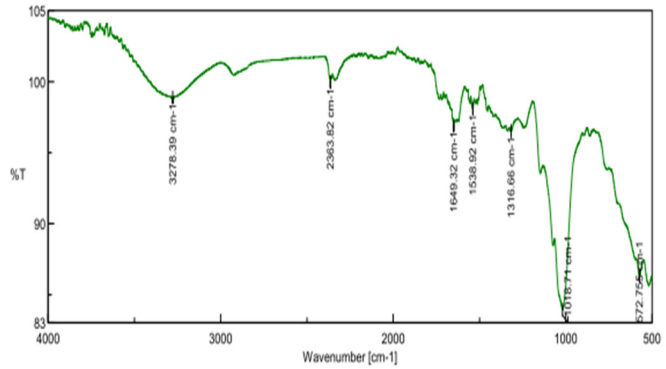

Figure 1. FTIR spectrum of cactus-derived pectin hydrogel

Based on the FTIR spectrum, with characteristic absorption peaks at 3278.39, 2926.82, 1649.32, 1538.02, 1316.66, 1076.11, and 872.72cm^{-1}, the following scientific interpretation can be made: The broad and intense peak at 3278.39cm^{-1} indicates the presence of O–H stretching vibrations, typical of strong hydrogen bonding, commonly observed in compounds rich in hydroxyl groups such as polysaccharides or water-bound materials. The broadness of this band reflects the complexity of hydrogen bonding interactions, either between sugar chains or between water molecules and organic structures. The peak at 2926.82cm^{-1}, within the C–H stretching region, is associated with the stretching vibrations of –CH_2– groups, confirming the presence of hydrocarbon chains or the polysaccharide backbone, especially from C–H bonds in sugar ring structures. In the region around 1649.32 cm^{-1}, the strong absorption band is commonly attributed to H–O–H bending of adsorbed water. Additionally, for polysaccharides containing uronic acid groups, the asymmetric stretching of carboxylate (–COO^-) also contributes to this band. The peak at 1538.02cm^{-1} may correspond to vibrational interactions involving S=O or C=O groups, indicating the potential presence of sulfated or carboxylated functionalities (Figure 1).

The absorption at 1316.66cm^{-1} represents skeletal vibrations of carbohydrate rings, including in-plane bending of C–H and ring deformation modes, which are characteristic of polysaccharide frameworks. The intense band at 1076.11cm^{-1} corresponds to the C–O and C–O–C stretching vibrations, particularly associated with glycosidic linkages between sugar monomers. This is a fingerprint region commonly seen in carbohydrate structures. The absorption at 872.72cm^{-1}, attributed to C–H out-of-plane bending vibrations, may indicate the presence of anomeric (α/β) configurations in carbohydrates or sulfated polysaccharide structures, especially those containing –OSO_3^- groups (Figure 1). This FTIR spectrum strongly supports the presence of a complex polysaccharide structure, rich in hydroxyl groups, containing glycosidic linkages, and showing evidence of sulfate and carboxylate functional groups.

The DSC (Differential Scanning Calorimetry) profile obtained from the cactus-derived pectin hydrogel clearly illustrates distinct physical and chemical phase transitions across specific temperature ranges. In the low-temperature region, from 59.72°C to a peak at 105.13°C, a pronounced endothermic peak is observed, with an enthalpy value of –7.477J/g. This phase corresponds to the physical dehydration process, including both free water and weakly bound water within the hydrogel network. Under the influence of heat, hydrogen bonds between water molecules and hydroxyl/carboxyl groups on the pectin chains are broken, leading to energy absorption an effect commonly observed in hydrophilic polysaccharides. In the temperature range of 151.19°C to 182.02°C, the profile displays two minor peaks at 151.19°C and 182.02°C, with corresponding enthalpy values of 3.371J/g and 1.411J/g (Figure 2).

These small thermal fluctuations represent minor transitions, possibly attributed to the glass transition (Tg) or local chain rearrangements within the polymer. At this stage, the polymer network remains structurally intact, but segmental motion begins, an important prelude to thermal softening and eventual degradation. The region between 222.00°C and 266.01°C marks the onset of initial degradation, with distinct exothermic peaks at 222.00°C (enthalpy: –0.891J/g) and 266.01°C (enthalpy: –5.865J/g). These peaks indicate demethylation processes, especially

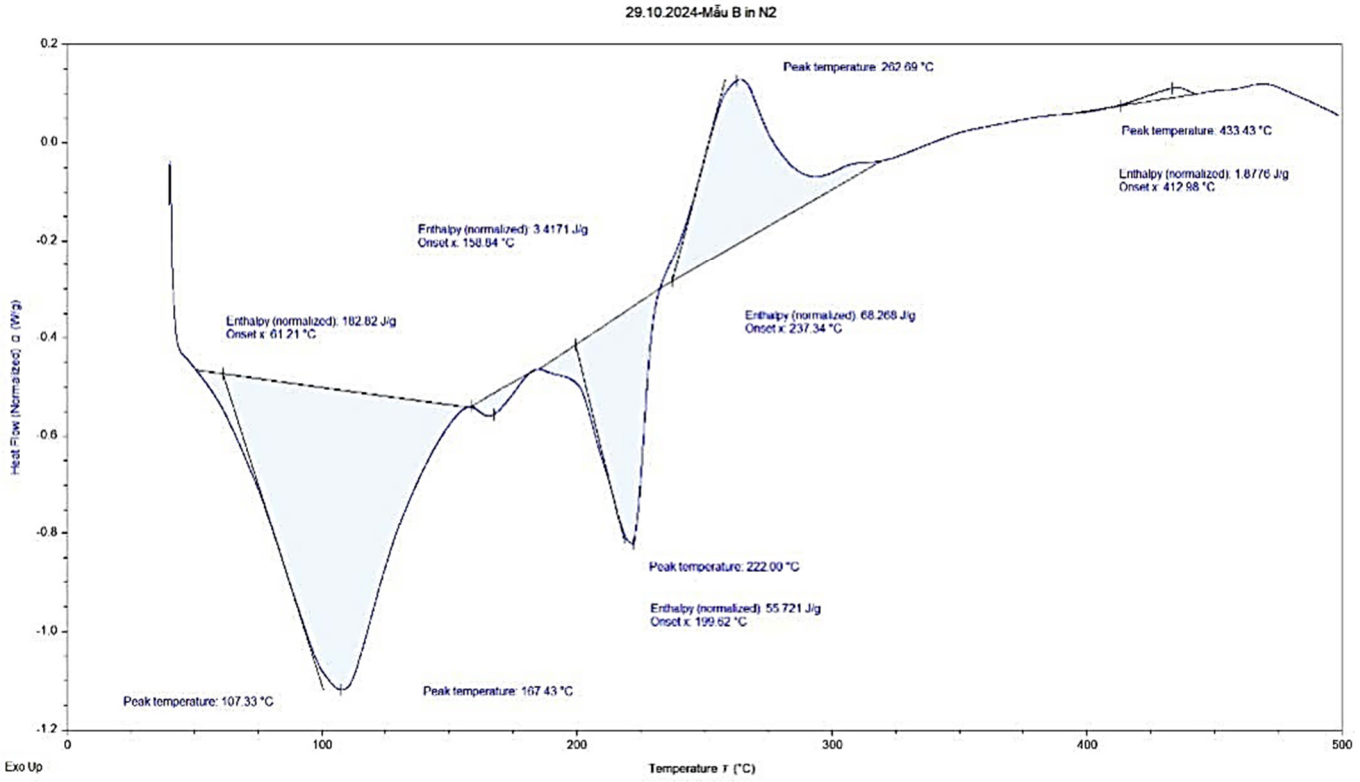

Figure 2. DSC profile of cactus-derived pectin hydrogel

in pectin segments with a high degree of esterification, and early chain scission. In addition, slight oxidative reactions or structural rearrangements may occur, leading to the release of energy. This stage is a critical turning point in the thermal decomposition of polysaccharides, signaling the onset of macromolecular breakdown of the hydrogel network. A major exothermic peak is observed at 407.92°C, with a significant enthalpy value of +167.60J/g (or 59.76J/g, depending on normalization method). This peak corresponds to the complete decomposition of the polysaccharide framework, characterized by the release of gaseous products such as CO_2 and H_2O, and the formation of carbonaceous residue and inorganic ash, including ions like Ca^{2+} or Mg^{2+} (Figure 2). This stage reflects the final phase of pyrolysis, typical of bio-based materials dominated by organic content.

The X-ray diffraction (XRD) pattern of the cactus-derived pectin hydrogel exhibits the typical characteristics of a semi-crystalline material. The presence of distinct diffraction peaks at 2θ angles of approximately 13°, 17°, 21°, 26°, and 29° (Figure 3), along with a broad underlying amorphous halo, reflects the nature of a natural polymer with inherent amorphous properties interspersed with crystalline mineral phases. This is consistent with the known properties of pectin, which has a flexible molecular structure and lacks long-range order, particularly when combined with minerals or metal ions (such as Ca^{2+}, Mg^{2+}...). The alternation between sharp peaks and broad diffuse scattering in the diffractogram clearly indicates the coexistence of an amorphous organic phase and an inorganic crystalline phase. It is likely that naturally occurring mineral crystals derived from cactus have become incorporated or interacted with the pectin matrix, thus imparting semi-crystalline properties to the material. A detailed analysis of the diffraction regions is summarized in Table 2.

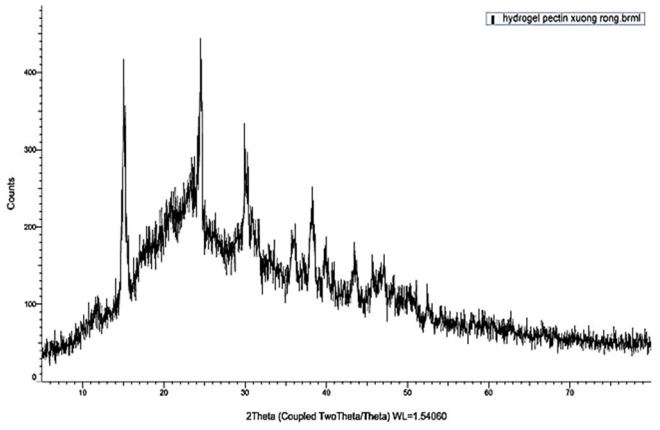

Figure 3. XRD pattern of cactus-derived pectin hydrogel

Table 2. Interpretation of the XRD pattern of cactus-derived pectin hydrogel

2θ Angle Range (°)	Peak Characteristics	Structural Interpretation
13 - 14°	Small peak	Indication of an amorphous structure from partially disordered pectin
16 - 18°	Distinct, sharp peak	Short-range ordering of pectin chains – characteristic of semi-crystalline behavior
20 - 22°	Broad cluster of peaks	Diffuse scattering from natural polymer matrix, indicating high amorphous content
25 - 27°	Sharp, high-intensity peak	Presence of crystalline mineral phases – possibly $CaCO_3$, MgO, or cactus-derived salts
28 - 30°	Series of small, successive peaks	Evidence of crystalline additives or mineral fillers embedded within the pectin matrix
> 30°	Gradually declining background	Amorphous organic phase lacking long-range crystalline order - typical of polysaccharides

In certain regions, large polymeric domains with wrinkled, layered structures and visible cracks are observed, indicating material shrinkage and phase separation following drying particularly after freeze-drying. The surface displays numerous microvoids, folds, and porous zones, which are characteristic of polysaccharides transitioning from hydrogel to dry form. Notably, discrete spherical microstructures with diameters of approximately 1 - 2μm, smooth surfaces, and clearly defined edges are evident. These structures are likely formed through colloidal aggregation or localized gelation, potentially driven by ionic cross-linking involving divalent cations such as Ca^{2+} present in the cactus extract. Their presence reflects the hydrogel's ability to self-organize into stable microparticles, indicating strong potential for applications such as drug encapsulation, delivery systems, or microstructured carriers. Additionally, clusters of spherical particles are randomly distributed across the polymer matrix, showing irregular sizes and rough, sometimes slightly deformed surfaces (Figure 4). This uneven distribution suggests that microparticles may form simultaneously during gelation, and are subsequently influenced by drying rate, surface tension contraction, or internal polymer network reorganization during dehydration. Cactus-derived pectin hydrogel not only forms a robust three-dimensional polymer network, but also simultaneously generates spherical microparticles that are well-distributed both on and within the matrix. The combination of a rough, porous gel matrix and stable spherical microstructures reflects an efficient gelation process and highlights the contributing role of natural mineral components from cactus extract in defining the structural and functional attributes of the material. The homogeneous porous matrix along with the appearance of spherical microstructures (1 - 2μm in diameter), which has not been previously described in Opuntia-derived pectin studies, indicates simultaneous gelation and microparticle formation. This suggests strong potential for use in bioactive compound encapsulation and delivery systems.

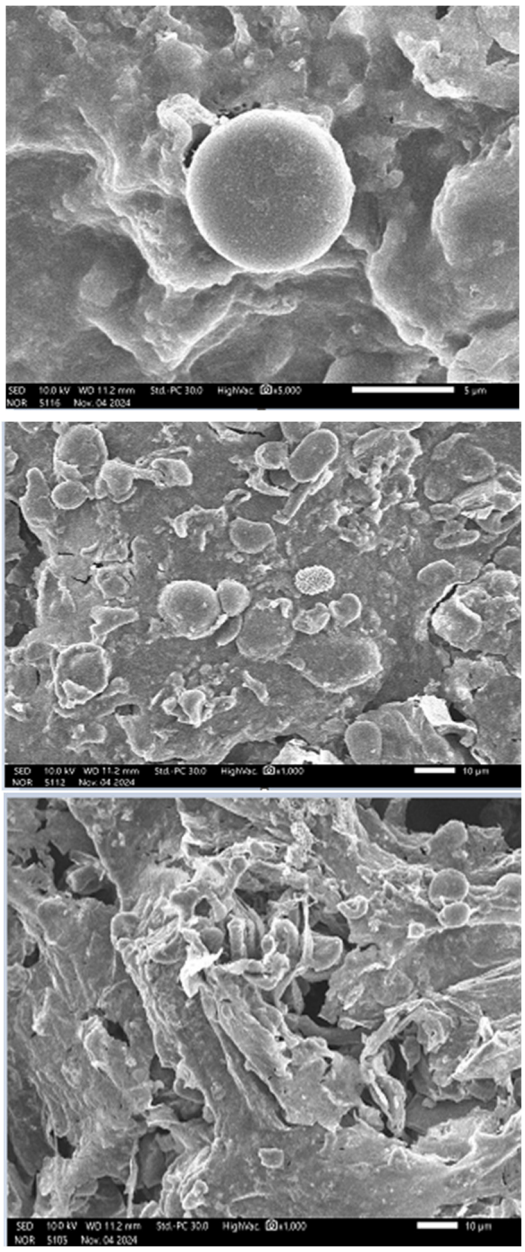

Figure 4. The scanning electron microscopy (SEM) image of cactus-extracted pectin hydrogel reveals a complex and diverse surface morphology, reflecting an effective gelation process and the influence of structural factors and drying conditions

The thermogravimetric analysis (TGA) profile of cactus-extracted pectin hydrogel reveals a distinct multi-stage thermal degradation pattern, reflecting the thermal characteristics of a natural polysaccharide-based system. The first stage occurs in the temperature range of 30 - 150°C, with an onset at 133.90°C. The sample shows a weight loss of 0.717mg, corresponding to 4.451%, which is attributed to the loss of free and weakly bound water within the hydrogel network. This phenomenon is commonly observed in hydrophilic polymer materials, as loosely hydrogen-bonded water molecules are released upon heating. The second stage also the most significant degradation phase occurs between 200 - 300°C, with an onset at 254.67°C and a peak at 293.49°C. During this stage, the sample undergoes a substantial weight loss of 8.202mg, accounting for 50.29% of its initial mass. This reflects the major decomposition of the pectin network, particularly involving the breakdown of methyl ester carboxyl groups and polygalacturonic acid chains, which constitute the main structural backbone of pectin. This stage is critical as it marks the thermal decomposition threshold of the material. The third stage occurs between 400 - 470°C, with an onset at 461.95°C. An additional 1.358mg, equivalent to 8.343%, is lost during this phase (Figure 5).

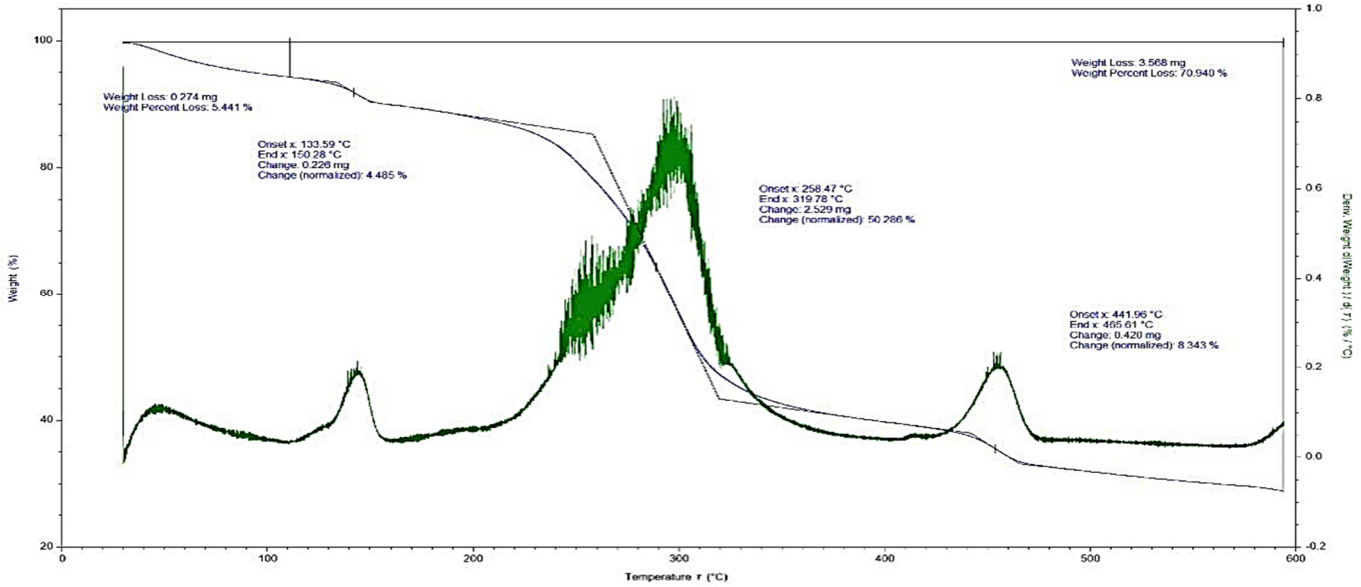

Figure 5. TGA profile of cactus-derived pectin hydrogel

It corresponds to the late-stage degradation of residual organic matter, potentially including residual carbon or mineral-organic compounds. This stage is characteristic of late pyrolysis, typically observed in bio-based materials containing natural mineral content. In total, the pectin hydrogel loses approximately 10.278mg, which represents 70.94% of the original mass. This indicates a near-complete decomposition of the organic material, leaving behind a residue likely composed of carbon char or thermally stable inorganic minerals, accounting for roughly 29% (Figure 5). The TGA profile confirms that the hydrogel exhibits a relatively low thermal stability, which is characteristic of natural polysaccharides. Moreover, the appearance of a high-temperature degradation stage highlights the influence of inorganic mineral components present in the cactus extract, contributing to the material's functional and structural enhancement. The clearly defined phase transitions and thermal resistance up to 407 °C provide evidence of a highly stable polymer network reinforced by ionic cross-linking with Ca^{2+} and STPP. This thermal behavior has not been specifically reported in prior studies of pectin hydrogels from *Opuntia dillenii*.

3.3. Biological activity of hydrogel

The results indicated that the obtained hydrogel exhibited promising antioxidant potential. The total antioxidant capacity (TAC) was measured at 0.29 ± 0.02mg ascorbic acid equivalent (AAE)/ml, reflecting the overall antioxidant performance of the hydrogel. The ferric reducing antioxidant power was recorded at 0.23 ± 0.01mg $FeSO_4$/ml, indicating the sample's ability to donate electrons in the reduction of Fe^{3+} to Fe^{2+}. The DPPH radical scavenging activity reached approximately 60% at a concentration of 1000ppm, confirming the hydrogel's antioxidant function via electron or hydrogen atom donation to neutralize DPPH radicals.

Statistical analysis revealed significant differences ($p < 0.05$) among hydrogel samples prepared under different

cross-linking conditions, suggesting that the concentrations of STPP and Ca^{2+} affected the antioxidant performance by altering the degree of cross-linking within the polymer network [9]. These findings are consistent with previous reports on hydrogels derived from natural pectin sources and reinforce the potential of this hydrogel for applications in functional foods and biomedical materials. This is among the few studies reporting distinct antioxidant activities (TAC ~0.29mg AAE/mL; DPPH ~60%) of hydrogels formed from *Opuntia* pectin in combination with STPP and Ca^{2+}, paving the way for the development of thermally stable, functionally bioactive natural hydrogels. Although control hydrogels were not included in this study, future comparative trials with common commercial pectin or alginate-based hydrogels are essential to further validate the superior functionality of the cactus-derived hydrogel.

4. CONCLUSION

This study is the first to report a comprehensive approach for extracting pectin from Opuntia dillenii using ultrasound-assisted extraction under optimized acidic conditions, yielding high-quality bioactive pectin with significant antioxidant capacity. The resulting hydrogel, formed via ionic crosslinking with STPP and Ca^{2+}, exhibits a novel combination of porous gel matrix and spherical microparticles, as confirmed by SEM. Spectroscopic (FTIR), thermal (DSC, TGA), and crystallinity (XRD) analyses collectively demonstrate the formation of a robust, semi-crystalline, thermally stable hydrogel.

The high antioxidant activity (~60% DPPH radical scavenging) and structural characteristics position this hydrogel as a novel biomaterial platform for applications in functional foods, active packaging, and drug delivery systems. Notably, this is among the few studies to combine cactus-derived pectin, dual ionic crosslinking, and full-spectrum physicochemical and bioactivity validation, offering a new direction for the sustainable valorization of local bioresources in Vietnam.

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