Synthesis of gel beads based on pectin extracted from dragon fruit stems and investigation of the effect of gel properties on the adsorption capacity of methyl orange

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ABSTRACT

In this study, polysaccharide gel beads (PS) extracted from dragon fruit stems were employed as adsorbents for the removal of Methyl Orange (MO) dye from aqueous solutions. The physicochemical properties of the PS were characterized by scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and Brunauer–Emmett–Teller (BET) surface area analysis. The influence of polymer concentration, pectin-to-alginate ratio, filler content, and gelation time on gel formation efficiency was systematically evaluated. Optimal gelation was achieved at a polymer concentration of 3%w/v, a pectin-to-alginate ratio of 1:1, a filler content of 60%w/v, and a gelation time of 60 min. Under these conditions, the PS demonstrated excellent adsorption performance, attaining a removal efficiency of 88.95% for MO after 120 min. These findings highlight the considerable potential of dragon fruit-derived polysaccharides for fabricating gel-based adsorbents applicable to the treatment of dye-containing wastewater.

Keywords: Pectin; Polysaccharide; Dragon fruit stem; Adsorption; Methyl orange.

1. INTRODUCTION

Dyes are widely used in textiles, paper, and biomedical industries due to their intense coloration and chemical stability [1]. Azo dyes, the most common synthetic colorants, are inexpensive, easy to synthesize, and structurally versatile, but they resist biodegradation and may degrade into toxic aromatic amines, posing risks to ecosystems and human health [1]. Even at low levels, dye contamination reduces light penetration and dissolved oxygen in water, highlighting the need for effective and eco-friendly removal technologies.

Methyl orange (MO), an anionic azo dye, is extensively applied in textiles, analytical chemistry, and printing. Its high solubility and stable structure allow persistence in water and resistance to conventional treatments [2]. Adsorption has emerged as an efficient remediation method, offering simplicity, cost-effectiveness, and high removal even at trace concentrations. Natural polymer-based adsorbents, particularly polysaccharide gels, show strong potential through electrostatic interactions with dye molecules [3].

Agricultural residues such as orange, apple, and banana peels, as well as dragon fruit stems, are promising sources of pectin [4, 5]. Valorizing these by-products both reduces waste and enhances economic value. Dragon fruit stems, rich in pectin with hydroxyl and carboxyl groups, are especially suitable for pollutant removal [6].

This study reports the fabrication of polysaccharide gel beads from dragon fruit stems and evaluates their performance in removing MO from water. The influence of polymer concentration, pectin-to-alginate ratio, filler content, and gelation time on gel formation was examined. Adsorption capacity and reusability were also assessed to explore sustainable dye removal applications.

2. MATERIALS AND METHODS

2.1. Chemicals

Pectin was extracted from dragon fruit stems and used without further purification [7]. Sodium Alginate (SA, 99.5%), Zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$, 99,0%), Methyl orange (MO, 99.8%) were obtained from Xilong, Chitosan (CS) was purchased from India and Activated carbon was purchased from Vietnam. Distilled water was used for dilution and solution preparation. All chemicals were analytical grade and used as received.

2.2. The synthesis of polysaccharide gel beads

Polysaccharide gel beads were prepared from a mixture of pectin, sodium alginate, chitosan, and activated carbon. The experimental conditions used to investigate the effects of polymer concentration, gelation time, the pectin-to-sodium alginate ratio, and filler content on the bead properties are summarized in table 1. The resulting solution was then dropwise added into a 4% zinc acetate dihydrate [$Zn(CH_3COO)_2.2H_2O$] solution to induce gelation and form spherical gel beads. The formed beads were subsequently soaked, thoroughly washed with distilled water, and dried to obtain the final gel bead product.

Table 1. *Summary of experimental conditions of PS preparation.*

Investigated parameter	Polymer concentration (%w/v)	Gelation time (min)	Pectin/Alginate Ratio	Filler content (%w/v)
Polymer concentration	1-5	60	-	-
Gelation time	3	1-120	-	-
Pectin/Alginate Ratio	3	60	1:1-1:5	-
Filler content	3	60	1:1	0-80

2.3. Adsorption experiments

Batch adsorption of methyl orange (MO) onto PS was performed in glass beakers by adding 0.2 g of PS to 50 mL of MO solution (50 mg/L). The mixtures were stirred at 200 rpm, and MO concentrations before and after adsorption were measured using a UV–Vis spectrophotometer (UV-1800) at 464 nm. The adsorption capacity at time *t* (q_t, mg/g) was calculated using the following equation:

$$q_t = \frac{V(C_o - C_t)}{m} \quad (1)$$

Where C_o and C_t are the initial and final dye concentration (mg/L), V is the volume of the dye solution (L), and m is the adsorbent mass (g).

The MO removal efficiency (%) was calculated using the following equation:

$$\%R = \frac{(C_o - C_t)}{C_o} 100\% \quad (2)$$

2.4. Reusability and regeneration studies

The reusability of PS gel beads was tested over five consecutive adsorption–desorption cycles using identical conditions to the initial experiments (50 mL MO solution, 50 mg/L, 120 min). After each cycle, the beads were washed with distilled water and ethanol for 30 min to remove residual dye, then reused with a fresh MO solution. Residual MO concentration was determined spectrophotometrically, and removal efficiency (%) was calculated using equation (2).

3. RESULTS AND DISCUSSION

3.1. Effect of polymer concentration

Polymer concentration is a key factor influencing gel formation (figure 1a). At 1% w/v, gel particles degraded rapidly within 6 minutes due to a weak, loose polymer network. Increasing the concentration to 2% extended the degradation time to 88 minutes, indicating greater structural stability. At 3%, the maximum stability of 179 minutes was achieved, suggesting this was the optimal concentration for gel formation. However, further increases to 4% and 5% reduced stability sharply to 62 and 61 minutes, respectively. This reduction likely arises from polymer chain compression and excessive aggregation, which decrease porosity and swelling capacity. Moreover, the increased viscosity limits Zn^{2+} penetration, restricting crosslinking within the gel core. As a result, the internal network remains sparse and weak, producing brittle gels prone to disruption in water [8].

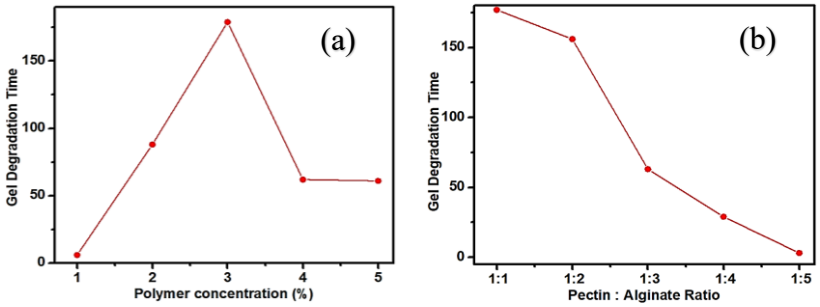


Figure 1. *Effect of (a) polymer concentration and (b) pectin/alginate ratio on the gelation characteristics.*

3.2. The effect of gelation time

Gelation time strongly affects gel strength in the early stages as the polymer network develops. As shown in figure 2b, extending gelation from 0 to 60 min increased destruction time from 4 to 186 min, indicating enhanced mechanical stability. Beyond 60 min, gel strength plateaued, suggesting saturation of network formation. Thus, 60 min is the optimal gelation time, balancing stability with production efficiency. At shorter times, incomplete crosslinking yields loosely connected networks. After 60 min, limited Zn^{2+} penetration into the core prevents further crosslinking, resulting in minimal structural change.

3.3. Effect of pectin/alginate ratio

The pectin:alginate ratio critically determines gel stability (figure 1b). As the alginate ratio increased from 1:1 to 1:5, gel degradation time sharply decreased from 177 to 3 minutes, highlighting alginate's key role in structural integrity. However, excess alginate without sufficient pectin reduced stability, as pectin strongly interacts with Zn^{2+} ions to reinforce the gel network [9]. Thus, a balanced 1:1 ratio provides optimal mechanical strength and structural stability.

When combined with pectin, alginate interacts to form a denser polymer network that enhances water retention and limits molecular mobility, thereby increasing viscosity. The polymer structure formation (PS) as a function of the pectin-to-alginate ratio is summarized in table 2.

Table 2. *The ability to form polysaccharide gel beads.*

Pectin: Alginate ratio	Properties
1:1	Large, spherical, and uniform gel beads.
1:2	Large, irregular gel beads.
1:3	Hard, irregular gel beads.
1:4	Poorly homogeneous mixture, high-viscosity solution, Irregular gel beads.
1:5	Poorly homogeneous mixture, high-viscosity solution, Irregular gel beads, and difficult gel formation.

At a pectin:alginate ratio of 1:1, the gel beads were large, spherical, and uniform. Increasing alginate disrupted bead stability, producing irregular and poorly formed structures. Hence, the 1:1 ratio was selected. The beads were then subjected to vacuum drying and freeze-drying to determine the most suitable method.

3.4. The effect of filler content

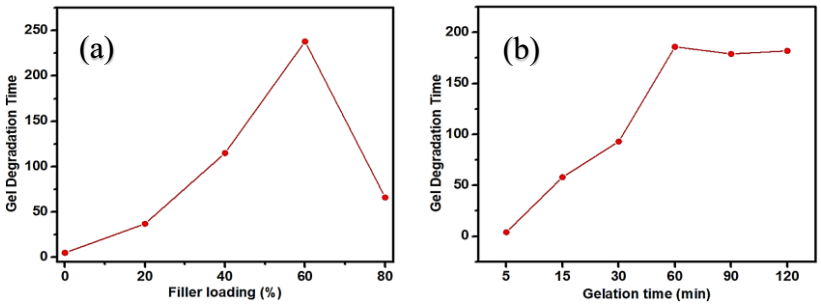


Figure 2. *Effect of (a) filler content and (b) gelation time on the gelation characteristics.*

The filler (chitosan and activated carbon) reinforced the gel network, regulated viscosity, and improved mechanical strength. As shown in figure 2a, increasing filler content from 0% to 60% w/v extended degradation time from 5 to 238 minutes, indicating enhanced stability. Uniformly dispersed fillers occupied pores, preventing collapse during drying and, together with the polymer matrix, forming a composite structure that stabilized pore walls. However, at 80% w/v, degradation time dropped to 66 minutes, likely due to network disruption from excess filler [10]. Thus, 60% w/v was identified as the optimal filler content for stable gel formation.

3.5. Characterization of polysaccharide gel

Pectin, alginate, and fillers (chitosan and activated carbon) were homogenized in water for 30 min, then dropped into 4% $Zn(CH_3COO)_2 \cdot 2H_2O$ to form gel beads, which were washed and dried under different conditions (figure 3).

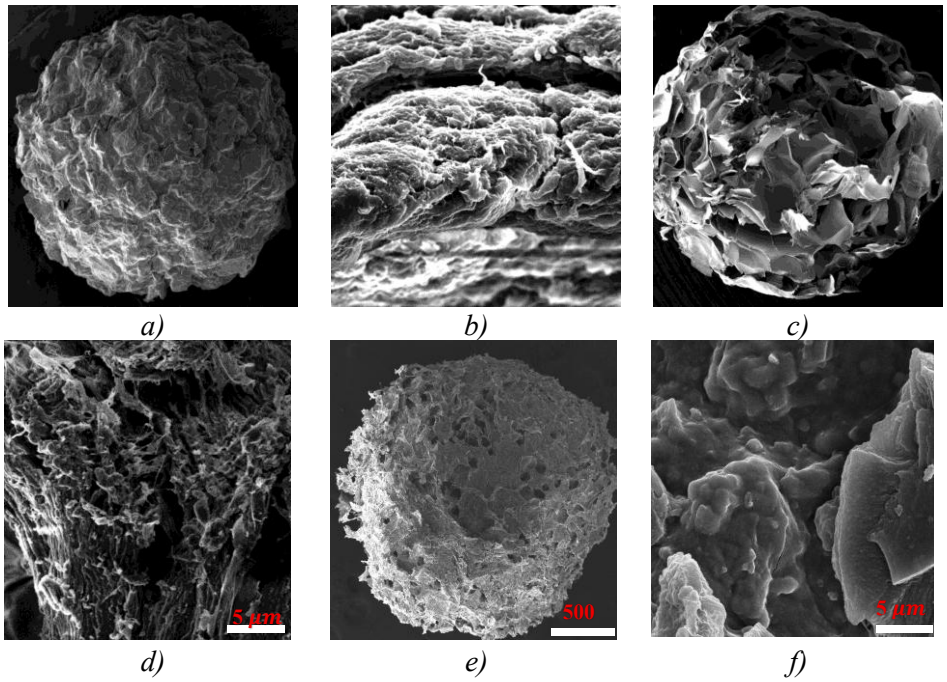


Figure 3. *SEM images of polysaccharide gel beads: (a–b) air-dried, (c–d) freeze-dried, and (e–f) after adsorption.*

Beads dried under ambient air (figure 3a–b) exhibited a dense, uniform outer layer, whereas freeze-dried beads (figure 3c–d) showed a porous structure with numerous voids, enhancing surface area, diffusion, and adsorption. After adsorption, the bead surface became homogeneous, with pores disappearing due to MO coverage (figure 3e–f). Nitrogen adsorption–desorption isotherms (BET) revealed a surface area of 103.69 m^2/g and an average pore diameter of 5.65 nm (BJH), classified as mesoporous by IUPAC. These mesopores, together with the large surface area, improve molecular diffusion and adsorption efficiency in aqueous systems.

The FTIR spectra of the polysaccharide gel (figure 4) show a broad band at ~3334 cm^{-1} (3689–3008 cm^{-1}), attributed to O–H stretching from intra- and intermolecular hydrogen bonds [12]. A distinct band at 2938 cm^{-1} corresponds to C–H stretching of CH, CH_2, and CH_3 groups [13]. In the fingerprint region, peaks at 1738 and 1611 cm^{-1} indicate C=O stretching of esters and N–H vibrations, while the 1500–1175 cm^{-1} region reflects C–H bending and C–N stretching [14]. Peaks at 1014 and 784 cm^{-1} are assigned to C–O stretching, typical of carbohydrates [15]. After MO adsorption, the O–H band shifted (3470–3669 cm^{-1}) with reduced intensity, suggesting disruption of internal hydrogen bonds and formation of new bonds with MO [16]. New MO-related peaks, such as $–SO_3$ at 1352 cm^{-1} and enhanced C=C at 1600 cm^{-1}, further confirm adsorption. Shifts of $–SO_3$, C–N, C–O, and aromatic ring vibrations indicate involvement of electrostatic forces, hydrophobic effects, and π–π interactions in MO binding [17].

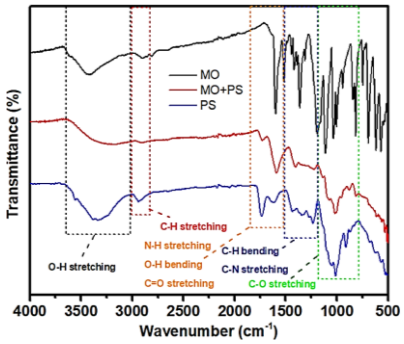
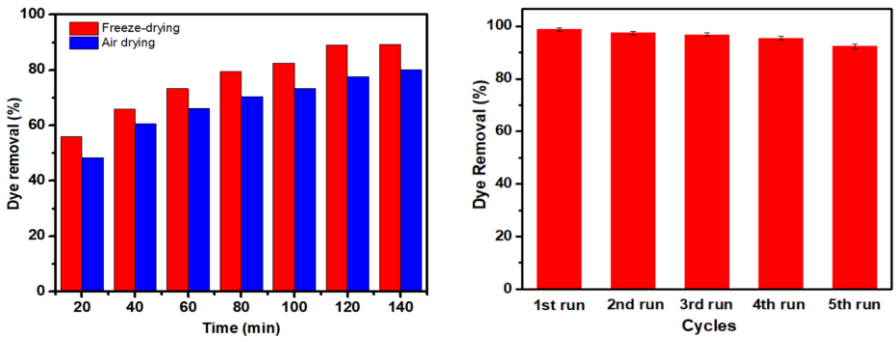


Figure 4. *FTIR spectra of the MO dye and after its adsorption by the Polysaccharide gel.*

3.6. Adsorption capacity of polysaccharide gel

The adsorption performance of the polysaccharide gel was investigated, revealing that the adsorption equilibrium was reached after 120 minutes, with a maximum removal efficiency of 88.95% and an adsorption capacity of 11.12 mg/g using the freeze-dried gel. The freeze-dried gel exhibited significantly higher adsorption efficiency compared to the air-dried counterpart. This enhanced performance is attributed to the porous architecture with abundant voids and internal cavities, as observed in figure 3 (c, d).



a) *b)*

Figure 5. *Adsorption capacity and reusability of PS.*

3.7. Reusability of adsorbents

The practical applicability of an adsorbent at the industrial scale largely depends on its reusability. In this study, the regeneration and reusability of the polysaccharide gel were evaluated following its application in methyl orange adsorption. As illustrated in figure 5b, the polysaccharide gel maintained an adsorption efficiency of 92.33% and an adsorption capacity of 10.27 $mg.g^{-1}$ after five consecutive cycles. The slight decrease in adsorption performance is attributed to incomplete desorption of the dye molecules. Nonetheless, these results demonstrate that the polysaccharide gel exhibits good regeneration and reusability potential.

4. CONCLUSIONS

In this study, polysaccharide gel beads (PS) from dragon fruit stems were prepared by dropping a polymer–filler mixture into 4% $Zn(CH_3COO)_2 \cdot 2H_2O$, forming spherical gels. The gels were dried by freeze-drying or air-drying, with freeze-dried PS showing a more porous morphology and superior adsorption. After 120 min, freeze-dried PS reached 88.95% MO removal efficiency and maintained high performance over five adsorption–desorption cycles. Compared with conventional adsorbents, the high efficiency, reusability, and low cost of PS underscore its potential for textile wastewater treatment.

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TÓM TẮT

Tổng hợp hạt gel trên cơ sở pectin tách chiết từ cây thanh long và nghiên cứu ảnh hưởng của tính chất gel đến khả năng hấp phụ methyl orange

Trong nghiên cứu này, hạt gel tổng hợp từ polysaccharide (PS) chiết xuất từ thân cây thanh long được sử dụng làm chất hấp phụ để loại bỏ thuốc nhuộm Methyl Orange (MO) khỏi dung dịch nước. Các đặc tính hóa lý của PS được đặc trưng bằng kính hiển vi điện tử quét (SEM), phổ hồng ngoại biến đổi Fourier (FTIR) và phân tích diện tích bề mặt theo phương pháp Brunauer–Emmett–Teller (BET). Ảnh hưởng của nồng độ polymer, tỷ lệ pectin-alginate, hàm lượng chất độn và thời gian tạo gel đến hiệu suất tạo gel đã được đánh giá một cách có hệ thống. Quá trình tạo gel tối ưu đạt được ở nồng độ polymer 3%w/v, tỷ lệ pectin–alginate 1:1, hàm lượng chất độn 60%w/v và thời gian tạo gel 60 phút. Trong điều kiện này, các hạt PS thể hiện hiệu suất hấp phụ vượt trội, đạt hiệu quả loại bỏ MO 88,95% sau 120 phút. Những kết quả này cho thấy tiềm năng đáng kể của polysaccharide có nguồn gốc từ cây thanh long trong chế tạo vật liệu hấp phụ dạng gel ứng dụng cho xử lý nước thải chứa thuốc nhuộm.

Từ khoá: Pectin; Polysaccharide; Thân cây thanh long; Hấp phụ; Methyl orange.