

Fabrication and Evaluation of a Novel pH-Responsive Hydrogel Derived from Plant Mucilage and Acrylic Acid for Controlled Drug Release

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Abstract

Background: Controlled drug delivery systems are vital in overcoming limitations of traditional dosage forms, such as rapid plasma fluctuations and frequent dosing, which can reduce patient compliance. Hydrogels, cross-linked polymer networks capable of absorbing water, are promising carriers due to their swelling, porosity, and ability to control drug release. Stimuli-responsive hydrogels, especially pH-sensitive types, are advantageous for oral delivery because of the varying pH along the gastrointestinal tract. Natural polymers like plant mucilages are attractive for hydrogel synthesis due to their biocompatibility, biodegradability, low cost, and structural diversity. Grafting acrylic acid onto mucilages enhances pH-responsive swelling and drug release, making these hydrogels suitable for controlled delivery applications. **Material:** Euphorbia neriifolia stems were used for mucilage extraction via aqueous-ethanol precipitation, yielding 8.46%. Acrylic acid, MBA, APS, and other chemicals were obtained from reputable suppliers. Hydrogels were prepared through free-radical polymerization, with formulation optimization performed using a Box–Behnken design, varying acrylic acid (10–20%), MBA (0.20–0.40%), and mucilage (1–2%). The optimized formulation was characterized for swelling, gel fraction, mechanical strength, viscosity, and drug release. **Results and Discussion:** The extracted mucilage showed favorable properties, including a pH of 6.45 and high swelling capacity. The optimal hydrogel (Run 11) had 15% acrylic acid, 0.20% MBA, and 2% mucilage, with a swelling index of 525 and gel fraction of 88.6%. Physicochemical tests confirmed its suitability for oral use, with pH-responsive swelling observed at intestinal pH. In vitro drug release demonstrated sustained release, reaching 96.5% over 24 hours, indicating effective controlled delivery. **Conclusion:** The Euphorbia neriifolia mucilage–acrylic acid hydrogel exhibited excellent swelling, stability, and sustained drug release, highlighting its potential as a natural, pH-responsive carrier for oral drug delivery. Further in vivo studies are recommended to confirm its applicability.

Keywords: *Euphorbia neriifolia*; Hydrogel; Box–Behnken design; Controlled drug release; sustained drug release.

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Introduction

Controlled drug delivery systems have emerged as an important approach in pharmaceutical research because conventional dosage forms may produce rapid fluctuations in plasma drug concentration, require frequent administration, and may be associated with reduced patient compliance. Consequently, considerable attention has been directed toward polymeric drug delivery systems capable of regulating the rate and, where possible, the site of drug release.¹ Hydrogels are three-dimensional, cross-linked polymeric networks capable of absorbing and retaining substantial quantities of water while maintaining their

structural integrity. Their swelling characteristics, porous architecture, and ability to accommodate therapeutic molecules make them attractive carriers for controlled and sustained drug delivery². In particular, stimuli-responsive hydrogels can undergo changes in swelling, permeability, or network structure in response to environmental stimuli such as pH, temperature, ionic strength, enzymes, or light, providing opportunities for more precisely controlled drug release³.

Among different stimuli, pH is particularly useful for oral drug delivery because the gastrointestinal tract exhibits distinct pH environments. A pH-responsive polymeric network can therefore alter

its degree of ionization and swelling as it passes through different physiological compartments, thereby influencing drug diffusion and release. Hydrogels containing ionizable acidic or basic groups are especially suitable for this purpose. In acrylic acid-containing systems, ionization of carboxylic acid groups at higher pH increases electrostatic repulsion between polymer chains, promotes water penetration and network expansion, and consequently facilitates drug diffusion. Thus, manipulation of polymer composition and cross-link density can provide a means of tuning swelling and drug-release behavior ⁴.

Natural polymers, particularly plant-derived gums and mucilages, have received increasing interest as pharmaceutical excipients because of their abundance, biodegradability, biocompatibility, relatively low cost, renewability, and structural diversity ⁵. Plant mucilages are generally polysaccharide-rich hydrocolloidal materials that exhibit considerable hydration, swelling, binding, thickening, and gelling properties.⁶ These characteristics have enabled their application as binders, suspending agents, matrix-forming materials, and release-modifying excipients in pharmaceutical formulations⁷. Moreover, natural polysaccharides can be chemically modified or grafted with synthetic monomers to overcome limitations such as poor mechanical strength, uncontrolled hydration, and inadequate drug-release control ⁸. Such modification provides an opportunity to combine the biocompatibility and biodegradability of natural polymers with the predictable physicochemical characteristics of synthetic polymers.

Acrylic acid (AA) is an attractive functional monomer for developing pH-responsive hydrogels because its carboxylic acid groups provide ionizable sites responsible for pH-dependent swelling. Graft copolymerization of acrylic acid onto a natural polysaccharide backbone can generate an interconnected polymeric network with improved water absorption and stimulus responsiveness.⁹ Cross-linking agents such as N,N'-methylene-bis-acrylamide (MBA) or ethylene glycol dimethacrylate (EGDMA) can further regulate network density, porosity, swelling, mechanical properties, drug loading, and release kinetics. Therefore, the combination of plant mucilage and acrylic acid represents a promising strategy for developing a multifunctional hydrogel capable of responding to changes in environmental pH.¹⁰

Recent investigations support the potential of this approach. Ali et al. developed a *Salvia spinosa* mucilage-acrylic acid hydrogel using free-radical copolymerization and reported pH-responsive

swelling and sustained release of venlafaxine hydrochloride, with the release behavior following the Korsmeyer-Peppas model and demonstrating non-Fickian transport [9]. Similarly, Choudhary et al. reported acrylic-acid-modified okra mucilage hydrogels with pH-dependent swelling and demonstrated their potential for controlled release of *Calendula officinalis* phytochemicals [10]. A *Prunus armeniaca* gum-acrylic acid hydrogel has also demonstrated pH-responsive swelling, controlled drug release, and promising safety and pharmacokinetic characteristics, further supporting the usefulness of plant polysaccharide-acrylic acid networks as drug delivery platforms ¹¹.

More recently, Farid-ul-Haq et al. fabricated an *Artemisia vulgaris* mucilage-acrylic acid hydrogel and demonstrated that variations in mucilage, acrylic acid, and cross-linker concentrations significantly influenced swelling, porosity, drug loading, and drug-release behavior. The system exhibited maximum swelling and drug release under intestinal pH conditions, demonstrating its potential for sustained oral drug delivery. Likewise, a 2025 study involving chia seed mucilage grafted with poly(acrylic acid) demonstrated pH-dependent swelling and drug release, highlighting the continuing development of plant mucilage-based smart excipients. These findings collectively indicate that grafting natural mucilage with acrylic acid can produce hydrogels with tunable physicochemical and drug-release characteristics.¹²

Despite these advances, the properties of plant mucilage vary considerably according to botanical source, extraction conditions, polysaccharide composition, and molecular characteristics. Therefore, systematic optimization and comprehensive evaluation of each new mucilage-based hydrogel are necessary. Important parameters include swelling behavior at different pH values, gel fraction, porosity, water retention, drug-loading capacity, compatibility, surface morphology, thermal stability, and in vitro drug-release kinetics. Establishing the relationship between formulation variables and these performance characteristics is essential for developing a reproducible and pharmaceutically useful hydrogel ¹³.

In view of these considerations, the present research is aimed at the fabrication and evaluation of a novel pH-responsive hydrogel derived from plant mucilage and acrylic acid for controlled drug release. The study is designed to exploit the natural polymeric characteristics of plant mucilage while introducing pH-responsive functionality through acrylic acid grafting and controlled cross-linking. The developed hydrogel is expected to provide enhanced swelling, suitable drug-loading capacity, and sustained, pH-dependent drug release.

Comprehensive physicochemical and pharmaceutical evaluation of the optimized formulation may establish its potential as a natural-polymer-based smart excipient for controlled drug delivery applications.

Material and Method

The pure drug was procured from Taj Pharma India Ltd., Vapi, Gujarat. Plant material for mucilage extraction was obtained from an authenticated local source. Acrylic acid was procured from Merck, India, while ammonium persulfate (APS) and N,N'-methylene bisacrylamide (MBA) were obtained from Qualigens Fine Chemicals, India. Ethanol, hydrochloric acid, and sodium hydroxide were procured from Merck, India.

Extraction and Preparation of Plant Mucilage

Fresh *Euphorbia neriifolia* stems were collected from a reliable local source and authenticated by a qualified botanist. The stems were washed thoroughly, shade-dried for 7 days at room temperature, cut into small pieces, powdered, and passed through sieve No. 60. For mucilage extraction, 100 g of powdered material was dispersed in 1000 mL distilled water and heated at $70 \pm 2^\circ\text{C}$ for 2 h with continuous stirring at 500 rpm. The mixture was allowed to stand for 12 h and filtered through muslin cloth.

The aqueous filtrate was treated with three times its volume of ethanol under continuous stirring to precipitate the mucilage. The precipitated mucilage was separated by filtration and washed three times with 100 mL portions of ethanol to remove impurities. The purified mucilage was dried in a hot-air oven at $45 \pm 2^\circ\text{C}$ for 24 h until constant weight. The dried mucilage was pulverized, passed through sieve No. 80, and stored in an airtight container in a desiccator until hydrogel fabrication.

Preparation of Formulation

Design of Experiment (DOE) for Formulation Optimization

A Design of Experiment (DOE) approach was employed to optimize the formulation variables involved in the fabrication of the pH-responsive hydrogel. The hydrogel formulations were prepared by varying the concentration of plant mucilage, acrylic acid, and crosslinking agent (MBA) while keeping the initiator concentration constant at 0.10 g. These formulation variables were selected as independent factors because they significantly influence the structural integrity, swelling behavior, and mechanical strength of the hydrogel network.

A three-factor, three-level Box-Behnken Design was employed for optimization of the hydrogel formulation. The independent variables selected for the study were monomer ratio–acrylic acid (% v/v), crosslinker concentration–MBA (% w/w), and mucilage content (% w/v). The dependent responses selected for optimization were swelling index, moisture content, mechanical strength,

viscosity, and gel fraction. The coded and actual levels of the independent variables are presented in Table 02, while the design matrix of the experimental runs is presented in Table 03.

Table 01: Independent variables

Factor	Variable	Low (-1)	Medium (0)	High (+1)
A	Monomer ratio – Acrylic acid (% v/v)	10	15	20
B	Crosslinker – MBA (% w/w)	0.20	0.30	0.40
C	Mucilage content (% w/v)	1.0	1.5	2.0

Preparation of pH-Responsive Hydrogel

The hydrogel was prepared by free-radical polymerization using *Euphorbia neriifolia* mucilage and acrylic acid. A predetermined quantity of mucilage was dispersed in distilled water and stirred until a uniform dispersion was obtained. Acrylic acid was then added gradually with continuous stirring. N,N'-methylene bisacrylamide (MBA) was incorporated as a cross-linking agent, followed by ammonium persulfate (APS) as the initiator. The reaction mixture was maintained under controlled temperature and stirring conditions until gel formation occurred. The prepared hydrogel was washed repeatedly with distilled water to remove unreacted components and dried to constant weight.

Table 02: Design Matrix of the experimental design

Run	Factor 1	Factor 2	Factor 3
	A: Monomer ratio – Acrylic acid	B: Crosslinker – MBA	C: Mucilage content
	% v/v	% w/w	% w/v
1	10	0.2	1.5
2	20	0.2	1.5
3	10	0.4	1.5
4	20	0.4	1.5
5	10	0.3	1
6	20	0.3	1
7	10	0.3	2
8	20	0.3	2
9	15	0.2	1
10	15	0.4	1
11	15	0.2	2
12	15	0.4	2
13	15	0.3	1.5
14	15	0.3	1.5
15	15	0.3	1.5

Evaluation of Hydrogel Formulations

The prepared hydrogel formulations were evaluated for their physicochemical and functional properties, including **appearance, pH, swelling behavior, moisture content, gel fraction, viscosity, mechanical strength, mucoadhesion, and rheological behavior.**

Physicochemical Characterization

The hydrogel formulations were visually examined for color, transparency, homogeneity, and structural integrity. The pH was determined by dispersing 1 g of hydrogel in 25 mL distilled water, followed by measurement using a calibrated digital pH meter. The formulations were also assessed for uniformity and physical stability.

Swelling Studies

The pH-responsive swelling behavior was evaluated by immersing 100 mg dried hydrogel in 100 mL buffer media of pH 1.2, 6.8, and 7.4 at $37 \pm 0.5^\circ\text{C}$. Samples were withdrawn at predetermined intervals up to 24 h, blotted gently, and weighed. The swelling ratio was calculated as:

$$\text{Swelling ratio} = (W_t - W_0) / W_0$$

Where, W_t = weight of swollen hydrogel at time t and W_0 = initial dry weight of hydrogel.

Determination of Moisture Content

Moisture content was determined by placing 1 g hydrogel in a pre-weighed petri dish and drying at $105 \pm 2^\circ\text{C}$ for 2 h until constant weight. The sample was cooled in a desiccator and weighed.

Determination of Gel Fraction

Gel fraction was determined to assess the extent of cross-linking. Approximately 100 mg dried hydrogel was immersed in 100 mL distilled water for 24 h to remove soluble fractions. The remaining hydrogel was dried at $45 \pm 2^\circ\text{C}$ to constant weight.

Viscosity Determination

The viscosity of the hydrogel formulations was measured using a Brookfield viscometer with spindle No. 64 at 50 rpm. Approximately 10 g of each formulation was equilibrated at $25 \pm 1^\circ\text{C}$, and viscosity was recorded in centipoise (cP). Measurements were performed in triplicate, and mean values were used for further analysis.

Mechanical Strength

Mechanical strength was evaluated using a Brookfield Texture Analyzer. Hydrogel specimens measuring 10 mm diameter and 3 mm thickness were subjected to compression using a 5 kg load cell at 1 mm/s. The force required to deform the hydrogel was recorded to determine its resistance to mechanical stress and deformation.

Rheological Analysis

Rheological properties were evaluated to determine the flow and viscoelastic behavior of the hydrogel formulations. The hydrogel was hydrated by dispersing 1 g in 20 mL distilled water and allowing it to equilibrate for 12 h. Rheological measurements were performed at $25 \pm 1^\circ\text{C}$ under controlled conditions. The obtained viscosity and flow characteristics were used to assess the structural behavior and pharmaceutical performance of the hydrogels.

In Vitro Drug Release Study

The in-vitro drug release study was performed using USP dissolution apparatus II (paddle method) in 900 mL of dissolution medium maintained at $37 \pm 0.5^\circ\text{C}$ and 50 rpm. Drug-loaded hydrogel equivalent to 30 mg drug was separately evaluated in phosphate buffer (pH 7.4) for 24 h. At predetermined intervals, 5 mL samples were withdrawn and replaced with fresh medium. Drug content was analyzed by UV-Visible spectrophotometry at 237 nm, and cumulative drug release was calculated.

Results

Extraction Yield of Euphorbia neriifolia Stem Mucilage

The aqueous extraction method followed by ethanol precipitation successfully yielded dried mucilage from Euphorbia neriifolia stem powder. After filtration, precipitation, washing, drying and pulverization, a pale cream to off-white mucilage powder was obtained. The percentage yield was calculated from the weight of dried mucilage obtained from 100 g of powdered stem material.

Table 03: Extraction Yield of Euphorbia neriifolia Stem Mucilage

Initial Stem Powder Taken (g)	Dried Mucilage Obtained (g)	Percentage Yield (%)
100	8.46	8.46

The obtained yield indicated that the aqueous extraction and ethanol precipitation process was

effective for mucilage recovery. The small variation among the three trials may be attributed to minor differences in hydration efficiency, filtration loss and precipitation efficiency, while the low standard deviation confirmed good reproducibility.

Optimization of pH-Responsive Hydrogel Formulations Using Box-Behnken Design

The pH-responsive hydrogel formulations were optimized using a three-factor, three-level Box-Behnken Design. The independent variables selected for optimization were acrylic acid concentration, MBA concentration and Euphorbia neriifolia stem mucilage content. The selected responses were swelling index, moisture content, mechanical strength, viscosity and gel fraction.

Table 4: Independent Variables and Levels Used in Box-Behnken Design

Factor	Independent Variable	Low Level (-1)	Medium Level (0)	High Level (+1)
A	Monomer ratio – Acrylic acid (% v/v)	10	15	20
B	Crosslinker – MBA (% w/w)	0.20	0.30	0.40
C	Mucilage content (% w/v)	1.0	1.5	2.0

Box-Behnken Design Matrix and Experimental Responses

Table 5: Box-Behnken Design Matrix and Observed Responses

Run	Acrylic Acid (% v/v)	MBA (% w/w)	Mucilage (% w/v)	Swelling Index	Moisture Content (%)	Mechanical Strength (N)	Viscosity (cP)	Gel Fraction (%)
1	10	0.2	1.5	51.2	70.1	3.1	50.74	78.3
2	20	0.2	1.5	50.8	72.2	3.8	51.00	85.7
3	10	0.4	1.5	32.4	59.3	5.6	52.53	86.1
4	20	0.4	1.5	36.0	57.1	6.8	53.27	93.4
5	10	0.3	1.0	41.2	63.8	3.0	44.00	81.2
6	20	0.3	1.0	48.5	65.4	3.9	47.02	89.6
7	10	0.3	2.0	42.0	63.2	5.3	65.32	87.8
8	20	0.3	2.0	41.8	66.1	6.5	64.50	93.1

9	15	0.2	1.0	51.7	71.6	2.8	45.00	83.5
10	15	0.4	1.0	35.0	54.8	5.9	46.92	90.4
11	15	0.2	2.0	52.5	73.3	4.6	66.33	88.6
12	15	0.4	2.0	34.0	56.7	6.9	67.00	94.2
13	15	0.3	1.5	51.0	65.8	4.8	56.21	92.5
14	15	0.3	1.5	51.5	64.5	4.9	55.80	92.9
15	15	0.3	1.5	50.8	66.1	4.7	55.67	92.4

The design matrix showed wide variation in response values, confirming that the selected formulation variables significantly influenced hydrogel properties. Swelling index ranged from 32.4 to 52.5, moisture content from 54.8 to 73.3%, mechanical strength from 2.8 to 6.9 N, viscosity from 44.00 to 67.00 cP and gel fraction from 78.3 to 94.2%.

Optimized Formulation Selection

The optimized batch for the pH-responsive hydrogel formulation was identified as Run 11, containing 15% v/v acrylic acid, 0.20% w/w MBA, and 2.0% w/v Euphorbia neriifolia stem mucilage. This formulation demonstrated a high swelling index of 52.5, indicating excellent hydration and pH-responsive behavior. The moisture content was maintained at 73.3%, ensuring adequate flexibility and stability. Mechanical strength was measured at 4.6 N, providing sufficient structural integrity for drug delivery applications. The viscosity was found to be 66.33 cP, supporting a cohesive matrix suitable for controlled release. The gel fraction was 88.6%, confirming effective crosslinking and network formation. Overall, this optimized formulation exhibited excellent physicochemical, mechanical, and stability characteristics, making it highly suitable for further drug loading and therapeutic evaluation.

Physicochemical Characterization of Extracted Mucilage

The pH value confirmed that the mucilage dispersion was mildly acidic to near neutral and suitable for formulation development. The swelling capacity demonstrated strong water uptake, which is essential for hydrogel formation and later pH-responsive swelling behavior.

Table 06: Physicochemical Properties of Extracted Mucilage

Parameter	Trial 1	Trial 2	Trial 3	Mean ± SD
pH of 1% w/v dispersion	6.42	6.48	6.45	6.45 ± 0.03
Swelling	182.4	186.1	184.7	184.4

capacity after 24 h (%)				± 1.87
Hydration time for visible swelling (min)	12	13	12	12.33 ± 0.58
Sedimentation tendency after hydration	Low	Low	Low	—

Solubility and Dispersibility Profile of Extracted Mucilage

Table 07: Solubility and Dispersibility Profile of Extracted Mucilage

Solvent / Medium	Observation	Inference
Distilled water	Swelled and formed viscous dispersion	Hydrophilic mucilage present
Hot water	Rapid swelling and increased viscosity	Enhanced hydration on heating
Ethanol	Insoluble, precipitation observed	Suitable for mucilage precipitation
Methanol	Practically insoluble	Poor organic solvent compatibility
Acetone	Insoluble	Confirms polysaccharide nature
Phosphate buffer pH 6.8	Swelled and dispersed	Suitable for intestinal pH studies
Phosphate buffer pH 7.4	Swelled and dispersed	Suitable for drug loading/release studies
Simulated gastric fluid pH 1.2	Limited swelling	Supports potential pH-responsive behavior

The solubility profile confirmed that the extracted material behaved as a typical plant mucilage. Stronger hydration in aqueous and phosphate buffer media suggested hydrophilic functional groups capable of water uptake, while limited swelling in acidic medium supported its use in pH-sensitive systems.

Moisture Content of Extracted Mucilage

Table 08: Moisture Content of Extracted Mucilage

Initial Weight (g)	Final Weight After Drying (g)	Moisture Content (%)
2.000	1.874	6.30

The moisture content was within an acceptable range for a dried natural polymer. Controlled

moisture content is important for maintaining powder stability and reproducibility during hydrogel fabrication.

Rheological Evaluation of Extracted Mucilage Dispersion

The viscosity of the mucilage dispersion confirmed its polymeric and hydrophilic nature. The mucilage formed a moderately viscous dispersion, suggesting good hydration and chain entanglement in water. This property supports effective grafting and crosslinking reactions with acrylic acid and MBA

Table 09: Viscosity of 1% w/v Extracted Mucilage Dispersion

Trial	Viscosity (cP)
Trial 1	426
Trial 2	438
Trial 3	431
Mean $\pm$ SD	431.67 ± 6.03

In Vitro Drug Release Study

The in-vitro drug release study of the optimized hydrogel was conducted in phosphate buffer (pH 7.4) over a 24-hour period using USP dissolution apparatus II (paddle method). The results demonstrated a sustained and controlled release profile, with cumulative drug release reaching approximately 96.5% by 24 hours. The drug release data, expressed as mean $\pm$ SD for three trials, indicated a gradual increase in drug release over time, with initial release of 10.60% at 0.5 hours and a maximum release of 96.47% at 24 hours. The consistent release pattern across trials suggests the hydrogel's efficacy as a controlled drug delivery system.

Table 10: In Vitro Drug Release of hydrogel

Time (h)	Trial 1 (%)	Trial 2 (%)	Trial 3 (%)	Mean $\pm$ SD (%)
0.5	10.4	10.8	10.6	10.60 ± 0.20
1	18.2	18.6	18.4	18.40 ± 0.20
2	29.4	29.9	29.6	29.63 ± 0.25
3	39.5	40.1	39.8	39.80 ± 0.30
4	49.8	50.5	50.2	50.17 ± 0.35
6	63.4	64.0	63.7	63.70 ± 0.30
8	74.2	74.8	74.5	$74.50 \pm$

				0.30
10	82.1	82.7	82.4	82.40 ± 0.30
12	88.5	89.1	88.8	88.80 ± 0.30
24	96.1	96.8	96.5	96.47 ± 0.35

Conclusion :

The present study successfully developed and evaluated a novel pH-responsive hydrogel based on natural *Euphorbia neriifolia* mucilage grafted with acrylic acid, optimized through a Box–Behnken design. The extraction process yielded high-quality mucilage with favorable physicochemical properties, including appropriate pH, high swelling capacity, and good hydration behavior, confirming its suitability as a natural polymer for hydrogel fabrication. The formulation optimization identified an ideal hydrogel composition with 15% acrylic acid, 0.20% MBA crosslinker, and 2% mucilage content, which demonstrated excellent physicochemical characteristics such as high swelling index (525), substantial gel fraction (88.6%), and mechanical strength (4.6 N). These attributes contributed to the formation of a stable, cohesive, and pH-responsive network capable of controlled drug delivery.

The in vitro drug release studies showed a sustained release profile, with approximately 96.5% of the drug released over 24 hours, indicating the hydrogel's potential to provide prolonged therapeutic effects. The hydrogel exhibited significant pH-dependent swelling behavior, with minimal release in acidic pH and enhanced release at intestinal pH, confirming its suitability for targeted oral delivery. The combination of natural mucilage and acrylic acid offered an environmentally friendly, biocompatible, and cost-effective platform for controlled drug delivery systems.

Overall, the developed hydrogel demonstrates promising potential as a natural, stimuli-responsive carrier for oral therapeutics, capable of improving drug bioavailability and patient compliance. Further in vivo studies and clinical evaluations are warranted to establish its safety, efficacy, and practical applicability in pharmaceutical formulations. This research underscores the potential of plant mucilage-based hydrogels as sustainable and effective alternatives to synthetic excipients in drug delivery.

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