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Review Paper

Hydrogel Beads as Novel Drug Delivery Systems: A Comprehensive Review

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ABSTRACT

Hydrogels are three-dimensional polymeric networks that can absorb a lot of water while preserving structural integrity thanks to cross-linking between polymer chains. Because of their distinct internal design, hydrogels have outstanding physicochemical and biological features such as high swelling capacity, biocompatibility, biodegradability, and controlled release capability. These qualities make them ideal for use in food technology, environmental protection, and biomedical disciplines. Hydrogel beads have received a lot of interest because of their spherical shape, large surface area, and effective encapsulation capacity for bioactive chemicals and pharmaceuticals. Hydrogel beads may be made from natural polymers including chitosan, sodium alginate, pectin, and gelatin, as well as synthetic polymers like polyvinyl alcohol and polyethylene glycol. Hydrogel beads are produced using a variety of manufacturing processes, including ionotropic gelation, emulsion polymerization, chemical crosslinking, radiation polymerization, and freeze-thaw procedures. Recent study has concentrated on chitosan-based hydrogel beads because of their biodegradability, antibacterial activity, and ability to absorb heavy metals. Hydrogel beads have demonstrated encouraging results in oral drug administration, mucosal distribution, transdermal systems, wound healing, and controlled release formulations. This paper describes the structure, categorization, polymer kinds, preparation techniques, and current advances in hydrogel bead technology, focusing on chitosan-based hydrogel systems.

INTRODUCTION

A hydrogel is a three-dimensional network structure produced by polymer polymerization or

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crosslinking[1]. Because of their unique internal design, hydrogels display little swelling in aqueous conditions while preserving structural integrity. These materials have various advantages, including high biocompatibility, biodegradability, and prospective use as drug delivery vehicles. As a result, hydrogels are being used in industries such as food, environmental protection, and medicine. Hydrogels may be made with a variety of materials and processes. Polysaccharides and proteins are the two most often utilized ingredients for hydrogel production, however other materials can be employed as well. This study discusses the methods for making hydrogels and hydrogel beads from polysaccharides (such as chitosan) and certain proteins, as well as the current advancements in research on chitosan-based hydrogel beads. In terms of production methods, hydrogels may be prepared in two ways: physically and chemically crosslinked. The physical crosslinking method largely depends on hydrogen bonding and electrostatic interactions between materials to produce polymeric networks, resulting in hydrogels[2]. A significant example is the use of electrostatic interactions between chitosan and sodium alginate, two polysaccharides, together with hydrogen bond formation to produce a polyelectrolyte complex. This method has been successfully used to create hydrogel beads containing Perilla frutescens L. essential oil. The investigations indicated that the compact structure of hydrogel beads could protect Perilla frutescens L. essential oil well, with the encapsulation effectiveness and loading rate of Perilla frutescens L. essential oil being 61.29 and 41.11%, respectively.[3] Furthermore, research has revealed the capability of creating hydrogels by combining polysaccharides and proteins. For example, a curcumin-encapsulating hydrogel was created by electrostatic interactions between gum Arabic (a polysaccharide) and gelatin (a protein)[4]. The results indicated that the produced hydrogel beads may increase curcumin's antioxidant activity, while also scavenging DPPH and ABTS free radicals at 95.59% and 87.65%, respectively. At the same time, hydrogel beads can effectively delay the impact of curcumin. In addition to electrostatic interactions and hydrogen bonding, freeze-thaw cycles can cause physical crosslinking. A practical use of this technology is to use polyvinyl alcohol and starch as basis ingredients and include black wolfberry anthocyanins into the solution. Researchers created a hydrogel that can detect the freshness of mutton using repeated freeze-thaw cycles. Furthermore, because to anthocyanins' natural antibacterial qualities, the resultant hydrogel efficiently increases the shelf life of mutton[5].

Because of their unusual spherical form and large specific surface area, hydrogel beads serve an important role in food, medicine, and the environment. The bead-like structure of hydrogels makes them more usable. Furthermore, the tiny size of hydrogel beads allows them to be included into food matrices without affecting the sensory quality of the meal. Hydrogel beads have received a lot of interest in modern culinary research because of their aesthetically attractive spherical shape. These beads typically have a diameter of millimeters. For example, some researchers have created hydrogel beads with diameters ranging from 3.79 to 4.23 mm[6], whilst others have created beads with diameters ranging from 2.6 to 5.6 mm[7]. Hydrogel beads may be made from a number of basic materials, the most common being chitosan (CS). CS is the second most common biopolymer on Earth, behind cellulose. It comes from the shells of shrimp and other crustaceans. The chemical structure of CS is well displayed. CS is a copolymer made up of repeated 2-amino-2-deoxy-D-glucopyranose units and remaining 2-acetamido-2-deoxy-D-glucopyranose units[8]. CS characteristics are influenced by its molecular

weight (typically 1-5 × 105 g/mol), purity, and degree of deacetylation. Acetylation degree refers to the ratio of N-acetyl-d-glucosamine units in CS to the total number of units, and the average acetylation degree of CS is less than 50%[9]. CS has good biodegradability, nontoxicity, biocompatibility, antimicrobial characteristics, and heavy ion adsorption capacity. It is also commonly accessible and reasonably priced[10]. As a result, CS is the most often used substance for producing hydrogel beads. CS is the only positively charged polysaccharide in nature, which distinguishes it from other naturally formed polymers. Researchers have undertaken significant experiments with CS. Despite their various benefits, hydrogel beads made with CS have some disadvantages, including uneven production, non-uniform diameters, a lack of process repeatability, and low mechanical strength and chemical resistance [11]. To solve these challenges, researchers frequently combine novel materials in precise proportions with CS to create CS-based hydrogel beads with improved performance. There are several ways for making CS-based hydrogel beads, including procedures that take use of the intrinsic characteristics of CS and chemical methods that use crosslinking agents to aid in hydrogel bead production. As science and technology improve, more new ways for making CS-based hydrogel beads are expected to emerge in the future.

Structure and Properties of Hydrogel Beads

Hydrogel beads are made of hydrophilic polymer chains that have been cross-linked to form a network structure. When immersed in water, they absorb liquids and grow while remaining structurally intact.

Key Characteristics

- High water absorption capability.
- Non-toxic and biocompatible.
- Controlled drug release capabilities.
  - Demonstrates pH-responsive swelling behaviour.
  - Biodegradability

Classification of hydrogel products

The hydrogel products can be classified on different bases as detailed below:

Classification based on source

Hydrogels can be classified into two groups based on their natural or synthetic origins [12].

Classification according to polymeric composition

The method of preparation leads to formations of some important classes of hydrogels. These can be exemplified by the following:

(a) Homopolymeric hydrogels are polymer networks made from a single monomer species, serving as a fundamental structural unit for any polymer network [13]. Depending on the monomer type and polymerization procedure, homopolymers can have a cross-linked skeletal structure.

(b) Copolymeric hydrogels consist of two or more monomer species with at least one hydrophilic component arranged in a random, block, or alternating configuration along the polymer network [14]

(c) Multipolymer Interpenetrating polymeric hydrogels (IPNs) are a type of hydrogel made of two independent cross-linked synthetic or natural polymer components in a network form. Semi-IPN hydrogel has two components: a cross-linked polymer and a non-cross-linked polymer [14,15].

Classification based on configuration

The classification of hydrogels depends on their physical structure and chemical composition can be classified as follows:

(a) Amorphous (non-crystalline).

(b) Semicrystalline: A complex mixture of amorphous and crystalline phases.
(c) Crystalline.

Classification based on type of cross-linking

Hydrogels can be divided into two categories based on the chemical or physical nature of the cross-link junctions. Chemically cross-linked networks have permanent junctions, while physical networks have transient junctions that arise from either polymer chain entanglements or physical interactions such as ionic interactions, hydrogen bonds, or hydrophobic interactions [15].

Classification based on physical appearance

Hydrogels appearance as matrix, film, or microsphere depends on the technique of polymerization involved in the preparation process.

Classification according to network electrical charge

Hydrogels may be categorized into four groups on the basis of presence or absence of electrical charge located on the cross-linked chains:

(a) Non-ionic (neutral).
(b) Ionic (including anionic or cationic).
(c) Amphoteric electrolyte (ampholytic) containing both acidic and basic groups.
(d) Zwitterionic (polybetaines) containing both anionic and cationic groups in each structural repeating unit.

Hydrogel-forming natural polymers include proteins such as collagen and gelatine and polysaccharides such as starch, alginate, and agarose. Synthetic polymers that form hydrogels are traditionally prepared using chemical polymerization methods.

Polymers Used in Hydrogel Beads

Polymers play a crucial role in the preparation of hydrogel beads because they determine the swelling behaviour, mechanical strength, drug loading capacity, and drug release profile. Hydrogel beads are typically prepared using natural, semi-synthetic, or synthetic polymers depending on the intended pharmaceutical application.

1. Natural Polymers

Natural polymers are widely used in hydrogel bead formulation due to their biocompatibility, biodegradability, and low toxicity.

(a) **Sodium alginate** is one of the most frequent polymers used in hydrogel beads. It's a natural polysaccharide derived from brown seaweed. In the presence of multivalent cations such as calcium chloride, alginate ionotropically gels to produce hydrogel beads.
(b) **Chitosan** is a biodegradable polymer derived from the deacetylation of chitin. It has mucoadhesive characteristics, which make it suitable for gastrointestinal medication administration.
(c) **Pectin** is derived from citrus fruits and apple pomace. It is commonly utilized in hydrogel beads developed for colon-targeted medication delivery since it may be destroyed by colonic bacteria.
(d) **Gelatin,** A natural protein polymer derived from collagen. It creates hydrogels by physical or chemical crosslinking.

2. Semi-Synthetic Polymers

Semi-synthetic polymers are chemically modified natural polymers that provide improved mechanical strength and stability.

Carboxymethyl Cellulose: Carboxymethyl Cellulose is a modified cellulose derivative used in hydrogel beads to enhance swelling and viscosity. Hydroxypropyl Methylcellulose is commonly used to control drug release due to its gel-forming ability.

3. Synthetic Polymers

Synthetic polymers are used to produce hydrogel beads with better mechanical strength, stability, and tunable drug release profiles.

- **Polyvinyl alcohol:** Polyvinyl alcohol is commonly utilized in hydrogel production due to its excellent film-forming characteristics and biocompatibility.
- **Polyethylene Glycol:** Polyethylene glycol is widely used to increase hydrogel flexibility and hydrophilicity.
- **Polyacrylamide:** Polyacrylamide, which generates highly water-absorbent hydrogels, is widely employed in biological applications.

Methods of Preparation of Hydrogel Beads

Hydrogel beads are produced by forming a three-dimensional cross-linked polymer network in the presence of a drug or active compound. Cross-linking may occur through ionic interaction, chemical bonding, polymerization, or physical interactions. The selection of method depends on polymer type, cross-linking mechanism, and desired structural characteristics of the beads.

1. Ionotropic Gelation Method

Ionotropic gelation is one of the most commonly used techniques for preparing hydrogel beads using ionic polymers such as Sodium Alginate.

Principle

The method is based on the interaction between negatively charged polymer chains and multivalent cations. When the polymer solution comes into contact with a solution containing cross-linking ions such as Calcium Chloride, ionic bridges are formed between polymer chains, resulting in gel bead formation.

Procedure

1. The polymer is dissolved in distilled water to prepare a homogeneous viscous solution.
2. The drug or active compound is dispersed or dissolved in the polymer solution under continuous stirring.
3. The resulting mixture is transferred into a syringe, burette, or dropping device.
4. The polymer–drug solution is added dropwise into an aqueous solution containing multivalent cations.
5. Upon contact with the cross-linking solution, rapid gelation occurs and spherical beads are formed.
6. The formed beads are allowed to remain in the cross-linking solution for a specific period to ensure complete gelation.
7. The beads are then separated by filtration or decantation.
8. The collected beads are washed with distilled water to remove excess cross-linking ions.
9. Finally, the beads are dried at room temperature or in a hot air oven to obtain stable hydrogel beads.

2. Emulsion Polymerization Method

In this method, hydrogel beads are produced through polymerization within dispersed droplets of an emulsion system.

Principle

A water phase containing polymer and drug is dispersed in a continuous oil phase to form a water-in-oil emulsion. Polymerization or cross-linking occurs within these droplets, leading to the formation of hydrogel beads.

Procedure

10. The aqueous phase is prepared by dissolving the polymer and drug in distilled water.
11. The oil phase is prepared using a suitable organic solvent along with an emulsifying agent.
12. The aqueous polymer solution is slowly added to the oil phase with constant stirring to form an emulsion.
13. Continuous stirring produces fine droplets of the aqueous phase dispersed in the oil phase.

14. A polymerization initiator or cross-linking agent is added to initiate the polymerization reaction.
15. Polymerization takes place within the dispersed droplets, forming hydrogel beads.
16. After completion of the reaction, the beads are separated by centrifugation or filtration.
17. The separated beads are washed several times with suitable solvents to remove residual oil and surfactants.
18. The purified beads are then dried to obtain the final hydrogel beads.

3. Chemical Cross-Linking Method

Chemical cross-linking involves the formation of covalent bonds between polymer chains using chemical cross-linking agents such as Glutaraldehyde.

Principle

Functional groups present in polymer molecules react with the cross-linking agent to form stable covalent bonds, resulting in a three-dimensional hydrogel network.

Procedure

1. The polymer is dissolved in a suitable solvent to obtain a uniform solution.
2. The drug is incorporated into the polymer solution with continuous stirring.
3. A predetermined amount of chemical cross-linking agent is added to the polymer solution.
4. The mixture is stirred continuously to allow the cross-linking reaction to occur.
5. As the reaction progresses, the polymer chains form a cross-linked network that produces hydrogel beads.
6. The formed beads are separated from the reaction mixture by filtration.
7. The beads are washed repeatedly with distilled water or suitable solvent to remove unreacted chemicals.
8. The washed beads are then dried under controlled conditions to obtain hydrogel beads.

4. Radiation Polymerization Method

Radiation polymerization involves the use of high-energy radiation to induce polymer cross-linking and hydrogel formation.

Principle

High-energy radiation produces free radicals within polymer molecules. These radicals initiate cross-linking reactions between polymer chains, forming a three-dimensional hydrogel structure.

Procedure

9. The polymer solution is prepared in distilled water or another suitable solvent.
10. The drug or active compound is dissolved or dispersed in the polymer solution.
11. The mixture is transferred into suitable moulds or containers for irradiation.
12. The system is exposed to high-energy radiation under controlled conditions.
13. Radiation generates free radicals within the polymer matrix.
14. These radicals react with neighbouring polymer chains to produce cross-links.
15. The cross-linking process results in the formation of hydrogel beads.
16. The beads are collected and washed if necessary.
17. Finally, the beads are dried to obtain the finished hydrogel bead formulation.

5. Freeze–Thaw Method

The freeze–thaw technique is a physical cross-linking method commonly used for hydrophilic polymers such as Polyvinyl Alcohol.

Principle

During freezing, ice crystals form within the polymer solution, concentrating the polymer chains in the unfrozen regions. Upon thawing, the polymer chains interact through hydrogen bonding

and crystallization, leading to the formation of a physically cross-linked hydrogel network.

Procedure

1. The polymer is dissolved in distilled water to prepare a homogeneous solution.
2. The drug is incorporated into the polymer solution under continuous stirring.
3. The mixture is poured into moulds or droplet-forming devices to obtain bead shapes.
4. The samples are placed in a freezer at low temperature for a specific period.
5. During freezing, phase separation and crystallization of polymer chains occur.
6. The frozen samples are then thawed at room temperature.
7. The freezing and thawing cycles are repeated several times to strengthen the hydrogel network.
8. After completion of cycles, hydrogel beads are obtained and collected.
9. The beads are dried or stored in hydrated form depending on the intended use.

Characterization of hydrogels

Generally, hydrogels are characterized for their morphology, swelling property, chemical structure and elasticity. The important features for characterization of hydrogels are as follows:

1. pH

The pH of hydrogels is tested using a digital pH meter. The pH meter must be calibrated before use.

2. Scanning Electron Microscopy (SEM)

SEM may reveal information about a sample's composition, surface topography, and other qualities including electrical conductivity. Magnification in SEM may be regulated over a range of up to 6 orders of magnitude, from around 10 to 500,000 times.[16]

Fourier Transform Infrared Spectroscopy

It is an effective approach for determining the chemical structure of a material. It is founded on the premise that a substance's fundamental components, i.e. chemical bonds, may be activated and absorb infrared light at chemical bond-specific frequencies.

Swelling measurement

There are three main ways for measuring swelling in hydrogels:-

Method A

This process involves immersing the dried hydrogel in deionized water for 48 hours at room temperature using a roller mixer. After swelling, the hydrogel is filtered through a stainless steel net with 30 meshes (681 μm). Swelling is calculated as follows.

Swelling = WS-Wd/Wd

Where Ws is the weight of the hydrogel in its swollen condition and Wd is the weight of the hydrogel when dry.

Method B

In a volumetric vial, the dry hydrogel (0.05-0.1g) was dispersed in an appropriate amount of water (25-30 ml) for 48 hours at room temperature. The mixture is then centrifuged to separate the water-bound substance and the unabsorbed water. The free water is removed, and the swelling may be quantified using Method A above.

Method C

In procedure C, the dried gel is submerged in deionized water for 16 hours at ambient temperature. After swelling, the hydrogel was filtered through a stainless-steel net with a mesh size of 100 (149 μm).

Swelling is calculated as follows:-

Swelling= C×100/B

Where,

C is the weight of hydrogel obtained after drying and B is the weight of the insoluble portion after extraction with water [17]

X-ray diffraction

Diffraction analysis determines if a material is crystalline or amorphous. It determines whether polymers keep their crystalline structure or are distorted throughout the processing pressurization process. Diffraction analysis is a prominent method for studying the morphological properties of hydrogels.

In -Vitro drug release study

Because hydrogels are swollen polymeric networks with drug molecules occupying their interiors, release studies are conducted to better understand the process of release over time. The parameters are compared with the standard plot to determine the equivalency of the medication solutions. [18]

Rheology

The viscosity of hydrogels is measured using a cone plate viscometer at 4ºC. This viscometer is extremely specific for determining viscosity. The viscosity is determined by a simple equation using the angle of repose, height, and length.

Spreadability study

The equipment consisted of a wooden block with a scale and two glass slides with a pan fixed on a pulley. Excess formulation was sandwiched between two glass slides, and a 100-gram weight was put on the upper glass slide for 5 minutes to compare the formulation and establish consistent thickness. Weight may be added, and the time it takes to separate the two slides was used to calculate spreadability.

S= (m × l) / t

Where S is spreadibilty, m is weight tied on upper slide, l is length of glass slide and t is time taken in seconds [19]

X-ray diffraction

X-ray diffraction is used to understand whether polymers retain their crystalline nature or they get deform during pressurization process.

Applications

1. Drug administration through the oral cavity

The drug is integrated into hydrogels and delivered to the oral cavity for local therapy of mouth disorders such as stomatitis, fungal diseases, periodontal disease, viral infections, and oral cavity malignancies.

2. Drug delivery in the gastrointestinal tract

The gastrointestinal tract is the most frequent route of medication delivery because to its ease of administration for compliance therapy and wide surface area for systemic absorption. Hydrogel-based devices, similar to buccal delivery, can be created to deliver medications to particular areas in the GI tract. For example, stomach-specific antibiotic medication delivery devices to treat Helicobacter pylori infection in peptic ulcer disease**.** [20]

3. Wound healing

Hydrogels may store water and drugs because of their cross-linked nature. Because of their propensity to keep water, they can hold and maintain wound exudates. When applied, gelatin and sodium alginate hydrogels can cover and protect wounds from bacterial infection.

4. Hydrogels for the brain

The blood-brain barrier, like other barriers in the human body, presents a problem for medication delivery, with 98% of newly synthesized medicines failing to penetrate it. As a result, there are very few medicines available for CNS medication delivery. Camptothecin, a long-term sustained release medication, is filled with PLGA microspheres, as shown in rats. These

microspheres extend the life time of rats with malignant gliomas. [21]

5. Rectal Delivery

It is generally understood that medications absorbed from the lower region of the rectum enter the systemic circulation immediately. Thus, the rectal route is effective for medication delivery due to first pass metabolism. Its principal application has been the local treatment of rectum-related disorders such as haemorrhoids.

6. Ocular medication delivery

Hydrogels are the most often utilized ocular medication delivery technique. Most hard and soft contact lenses are made of polymers in the form of hydrogel films. In-situ forming hydrogels are appealing as an eye drug delivery method because of their ease of dosing as a liquid, and their long-term retention characteristic as a gel following dosing. [22]

7. Subcutaneous Delivery

Hydrogels are biodegradable in nature; by using this trait, we may create biodegradable implantable hydrogels. Cross-linked PHEMA is being produced for use in subcutaneous administration of anticancer medicines, specifically cytarabine.

8. Transdermal Delivery.

Various hydrogel-based drug delivery devices are developed to administer drugs via the transdermal route. Swollen hydrogels can serve as controlled release devices in the field of wound dressing. Hydrogel-based formulations are being investigated for transdermal iontophoresis to improve penetration of drugs like as hormones and nicotine.

9. Topical medication delivery.

Hydrogels have been utilized to deliver active ingredients such as Desonide, a synthetic corticosteroid often used as an anti-inflammatory. The hydrogels have been developed to improve patient compliance and contain moisturizing qualities, thus scaling and dryness are not predicted with this drug delivery technology. [23]

CONCLUSION

Hydrogel beads are a significant family of polymeric materials with numerous uses in pharmacological, biological, food, and environmental sciences. Their three-dimensional cross-linked structure allows them to absorb large amounts of water while remaining stable, making them effective transporters for medicines and bioactive chemicals. Hydrogel beads may be made from a number of natural, semi-synthetic, and synthetic polymers, with chitosan and sodium alginate being among the most extensively researched. Different preparation procedures, including as ionotropic gelation, emulsion polymerization, chemical cross-linking, radiation polymerization, and freeze-thaw processes, allow for the creation of hydrogel beads with a variety of structural and functional features. In recent years, major research has concentrated on enhancing the mechanical strength, stability, and homogeneity of hydrogel beads via polymer mixing, chemical modification, and new production methods. Despite several constraints, such as particle size variability and mechanical strength, hydrogel bead systems are evolving with the introduction of new polymers and cross-linking techniques. Future studies are expected to improve the performance of hydrogel beads and broaden their uses in targeted drug delivery, tissue engineering, and smart biomedical systems.

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