J Nanostruct 16(4): 4588-4595, Autumn 2026

RESEARCH PAPER

A Novel Nanocomposite Hydrogel Designed for Biomedical Applications and Synthesized Using Polyvinyl Alcohol, Chitosan, and SiO_2: Formulation and Morphological Characterization

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ARTICLE INFO

Article History:
Received 03 May 2026
Accepted 13 July 2026
Published 01 October 2026

Keywords:
Morphology
Porosity
Swelling Capacity
Water uptake

ABSTRACT

Nanocomposite hydrogels (NCHGs) have recently emerged as promising materials for different biomedical applications, including cellular therapy, wound dressings and controlled drug delivery due to their high-water absorption capacity and biocompatibility. NCHGs are three-dimensional, interconnected and swellable polymer. This work aims to synthesize NCHGs with varying ratios of chitosan /polyvinyl alcohol polymers and functionalized with silica to improve structural integrity and morphological characteristics, and to characterize it and evaluate its swelling in physiological environments. The NCHG was developed by controlled blending and crosslinking techniques, ensuring homogeneous dispersion of SiO_2 nanoparticles throughout the polymer matrix. The characterization included Fourier-transform infrared spectroscopy, which confirmed the successful integration of components, revealing characteristic interactions between the hydroxyl and amine functional groups of the polymers and the silica. While X-ray diffraction indicated the successful incorporation of nanoparticles without compromising the structural integrity of the polymer network. Moreover, scanning electron microscopy revealed a rough and porous morphology, providing an effective scaffold for nanoparticle incorporation. The results of swelling showed controlled behaviour that enhance high swelling in acidic environments as in tumor, and retain its stability in neutral environments. In conclusion, the developed NCHG suggests its significance in various medical applications and including drug loading and control release, due to their highly porous, strong water absorption capacity and controlled swelling behaviour.

How to cite this article
Najeh Chfat K, Hadi Aysa N, Al-Azzawi S. A Novel Nanocomposite Hydrogel Designed for Biomedical Applications and Synthesized Using Polyvinyl Alcohol, Chitosan, and SiO_2: Formulation and Morphological Characterization. J Nanostruct, 2026; 16(4):4588-4595. DOI: 10.22052/JNS.2026.04.008

INTRODUCTION

Hydrogels are intricate, interconnected, water- attracting structures that can absorb large amounts of water while being insoluble in it [1]. They have been accreted using a wide variety of natural and synthetic polymers. Hydrogels represent a remarkable choice for use in health care applications, including cellular therapy,

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tissue regeneration, wound dressings, hygiene items, and regulated drug delivery [2]. Owing to their exceptional ability to retain water and their suitability for living organisms. Interconnected, three dimensional, expanding networks that utilize nanoparticles for wound dressings, hygiene pads, dental materials, and cellular therapy are known as nanocomposite hydrogels (NCHG), which can consist of different polymers like polyvinyl alcohol and chitosan [3]. Polyvinyl alcohol (PVA) is widely recognized as a safe and biocompatible material because of its the stability and lack of reactivity. Due to presence of OH groups on its chains allows interaction with different synthetics and natural compounds [4]. Through various physical and chemical methods PVA can produce a hydrogel. PVA hydrogels have generated significant interest because of their transparency, non-toxic nature, biocompatibility, and absence of carcinogenic elements. Consequently, application for lens implants, artificial heart coatings, wound coverings, drug delivery systems and synthetic cartilage in orthopaedic procedures fall under the classification of pharmaceuticals and biomaterials [5]. Most applications in both science and industry utilize nanoparticles (NPs). One such NPs is silica (SiO_2) which has various applications, such as drug delivery, sensing materials, electronics and as a catalyst [6, 7]. Silica nanoparticles possesses remarkable potential in drug delivery because of their adjustable particle and pore sizes, extensive surface area and alterable surface chemistry. These characteristics enable the adsorption and retention of a wide variety of therapeutic active compounds, encompassing small molecules and biomacromolecules. SiO_2NPs, because of their unique antibacterial properties, are often used in hospitals to eliminate more than 99% of bacteria within the initial two hours of contact [8]. Moreover, it has been found that SiO_2NPs are non-harmful to humans when used topically in a small amount, possess potent antifungal characteristics, and positively impact the skin [5, 6, 9, 10]. The regulation of drug release has been studied with different biomaterials, with hydrogels being among them because of their minimal drug interaction and facilitated drug diffusion into the hydrogel [2, 11]. Meanwhile, chitosan (CS) plays a significant role in the development of the NCHGs because of its unique physicochemical and biological properties. As a natural, biocompatible, and biodegradable polysaccharide, CS enhances the suitability of the hydrogel system for biomedical and drug delivery applications. It is suggested that, CS contributes to the development of a stable network of hydrogel through intermolecular H-bonding and electrostatic reactions with PVA. These interactions improve the mechanical properties and structural stability of the hydrogel, especially under aqueous or physiological conditions. Additionally, CS has amino groups that are positively charged, and this charge plays a significant role in drug loading and retention, especially when the drug has a negative charge

Fig. 1. The prepared hydrogel.

or is polar in nature. The addition of CS improves the ability of the hydrogel matrix to attract drug molecules due to electrostatic interactions as well as hydrogen bonding. The addition of CS also improves the pH-responsive properties of the hydrogel system. Due to the protonation/deprotonation properties of the amino group, changes in pH can control the swelling behavior of the hydrogel [12]. This property provides specific benefits to controlled drug release applications due to the ability to increase the rate of drug release under acidic conditions, which are generally related to tumor tissues. In addition, the addition of CS to the hydrogel increases hydrophilicity and swelling ability, thus facilitating the movement of drug molecules into and out of the polymeric structure [9]. The natural nature and low toxicity of CS make it suitable as a functional component of smart drug delivery systems. In conclusion, CS plays a role as a multifunctional component of the PVA/CS/SiO_2 hydrogel system, providing benefits to biocompatibility, efficiency of drug loading, pH sensitivity, and controlling the rate of controlled drug release [2, 3]. In previous studies, CS and PVA hydrogels have been investigated individually so the CS is known for it is biocompatibility while PVA is known for it is good mechanical and chemical stability [13, 14]. This study aims to address the limitations faced by single-component hydrogels with regards to low drug loading efficiency, uncontrolled release of the drug, and lack of mechanical integrity [15]. While much literature has already examined the hydrogel systems with regards to the delivery of drugs, there still seems to be a gap in the development of the hydrogel system that can simultaneously exhibit high mechanical durability, ultra-sensitive pH-sensitivity, and high drug loading efficiency. This study is significant as it aims to develop a series of hydrogels made from the polymer blend of PVA/CS/SiO_2 NCHGs with precisely tuned synergistic interactions between the organic polymers and the inorganic filler. Unlike other hydrogels, the addition of the inorganic filler SiO_2 to the hydrogel formulation does not only enhance the hydrogels mechanical characteristics but also boosts the cationic properties of the chitosan polymer backbone. This hydrogel system can attain high swelling in acidic environments, which can then facilitate the controlled burst release of the chemotherapeutic agent at the target tumor site and retain its stability in neutral environments. SiO_2 NPs are known to enhance the hydrogel system with regards to its structural stability, mechanical properties, swelling ratios, and the efficiency of the hydrogel system with regards to the loading of the drug [16]. As such, the NCHG system designed in this work is the culmination of the properties of each component of the hydrogel system [17].

This study was aimed to develop a series of PVA/CS/SiO_2 hydrogels with varying concentrations of PVA with maintaining constant amounts of CS and SiO_2 which might facilitate the loading and the controlled release of loaded drugs. In addition to study the physical appearance, homogeneity, and stability of the prepared hydrogel formulations, and to determine the swelling behaviour of the prepared hydrogels under acidic and neutral conditions.

MATERIALS USED AND METHODS

Materials used

The main synthetic polymer used was PVA, obtained from Hi Media Laboratories (india) and purity reported by supplier (typically ≥98%), with an average molecular weight in the range 85,000–

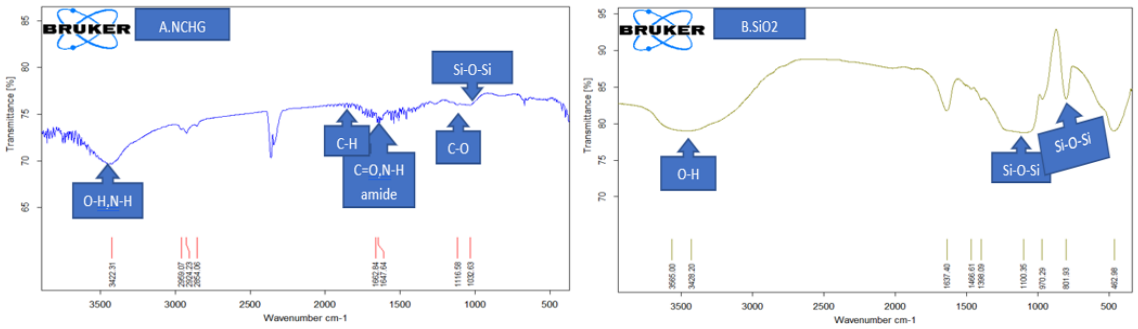

Fig. 2. FT-IR spectra of A: nanocomposite hydrogel (NCHG), showing characteristic peaks corresponding to polyvinyl alcohol, and chitosan. The broad band at 3420 cm^{-1} was attributed to overlapping O-H and N-H stretching vibrations, indicating strong hydrogen bonding within the hydrogel network and B: silica nanoparticles (SiO_2 NPs) in hydrogel NCHG, the peak observed at around 1087 cm^{-1} correspond to the asymmetric stretching vibration of Si-O-Si bonds, confirming the formation of silica network. While the peak overlapping confirms the successful incorporation of SiO_2 nanoparticles into the hydrogel matrix through intermolecular interactions.

124,000 g/mol, and a hydrolysis degree of 98–99%. The choice of synthetic polymer was based on the excellent water solubility, biocompatibility, and film-forming ability of PVA. The natural polymer used was CS, obtained from Macklin Biochemical (china) and purity reported by supplier (typically ≥95%), with low molecular weight in the range 150–400 kDa and a deacetylation degree of at least 75%, to enhance biocompatibility and pH sensitivity. Glacial acetic acid was purchased from Maharashtra (India) with purity (typically ≥99%). CS was dissolved in 1% glacial acetic acid aqueous solution prior to use. (SiO_2) with an average particle size in the nanometres range (100 nm) were used as an inorganic nanofiller to improve the structural stability and physicochemical properties of the hydrogel matrix. Acetic acid (glacial, analytical grade) was used for the preparation of chitosan solutions. Phosphate buffered saline (PBS, pH 7.4) was obtained from Maharashtra (India) with purity (typically ≥99%), was used as the swelling medium. Deionized water was used throughout the experiments for solution preparation. All chemicals were of analytical grade and were used without further purification.

The Preparation of PVA/CS Hydrogel

Aqueous solutions of PVA were prepared at different concentrations (5%, 10%, and 15%, 20% w/v). Briefly, the required amount of PVA powder was gradually added to deionized water under continuous stirring and heated at 80–90 °C until a clear and homogeneous solution was obtained. The solution was then allowed to cool to room temperature [18].

CS solution was prepared at different concentrations (0.5%, 1%, 2%) by dissolving it in a 1% (v/v) glacial acetic acid aqueous solution with magnetic stirrer at 40 °C until clear solution was obtained [19].

For the PVA/CS blend preparation, the solution of PVA was mixed with the CS solution at a fixed ratio under continuous stirrer at room temperature to obtain a homogenous polymeric mixture, ensuring uniform distribution of both polymers within the hydrogel matrix [20].

Weighted amounts of SiO_2 was then added gradually to the PVA/CS blend with continuous stirrer to obtain a homogeneous PVA/CS/SiO_2 nanocomposite mixture [21].

The resulting PVA/CS/SiO_2 mixture was casting into mold and petri dishes and permitted to cool and then placed in a refrigerator for 24 hours, followed by thawing for 4 hours at room temperature. This cycle of freeze-thaw was repeated for 12 cycles to improve cross-linking and mechanical interactions between PVA and CS until gelation occurred. The formed hydrogels (Fig. 1) were then left to stabilize completely prior to further characterization [22].

Characterization technique

A Fourier transform infrared (FTIR) spectroscopy on a Brucker vector was used to identify the functional groups within the hydrogel, as well as for SiO_2. The sample was finely ground with potassium bromide (KBR) and pressed to make pellets before FTIR spectroscopy. After that, the spectra were examined in transmission mode from 400 cm−1 to 4000 cm−1 while at same time recording the materials' X-ray diffraction pattern (=1.5048) to confirm the hydrogel formation. Scanning Electron Microscopy (SEM) was then utilized to investigate the NCHG morphology, to provide information on pore structure, porosity, and fiber characteristics.

Swelling evaluation of hydrogel

The hydrogels Equilibrium Swelling (ES) was

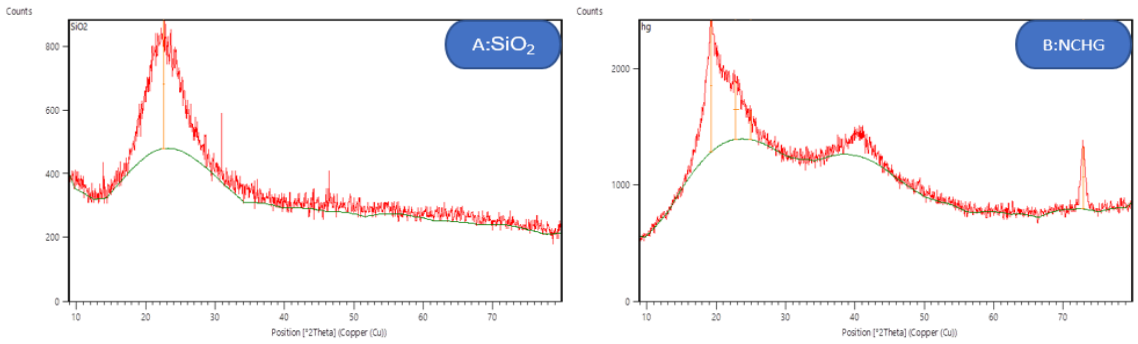

Fig. 3. The X-Ray Diffraction analysis of A: silica nanoparticles (SiO_2 NPs), and B: nanocomposite hydrogel (NCHG), exhibiting characteristic diffraction peak of SiO_2 and confirming its integration into hydrogel matrix. The broad diffraction pattern observed for the hydrogel indicates it is partially amorphous nature. The characteristic semi- crystalline peak of polyvinyl alcohol (PVA) at 2θ= 19 - 20 and the broad amorphous of SiO_2 indicate optimum nanoparticle dispersion within NCHG network.

assessed in different physiological solutions, including distilled water and PBS. To reach fullest capacity of swelling, 0.25 g of NCHG sample was soaked in 100 mL of distilled water and PBS (pH 5.5, pH 7.4) for 48 hours. After determined time intervals, swelling ratio was calculated by Eq. 1.

$$\text{swelling}\% = \frac{(W2 - W1)}{(W1)} \times 100 \quad (1)$$

Where (W1) is the dried sample weight of and (W2) is the swollen sample weight after 48 hours. The mechanical integrity and swelling behavior of the NCHG are critical parameters for evaluating its performance as a drug delivery vehicle.

Other mechanical properties of hydrogel such as tensile strength, elongation at break, young modulus and resilience, where evaluated by using universal testing machine. These parameters are commonly used to assess the strength, elasticity and structural integrity of hydrogel [23].

RESULTS AND DISCUSSION

FTIR analysis

Spectrometer results of FT-IR revealed that amino and hydroxyl groups:) Fig. 2A) The broad band at 3420 cm^{-1} is a composite of both O-H stretching and N-H stretching vibrations from the CS backbone. This supports the presence of strong intermolecular hydrogen bonding in the hydrogel. The presence of amide bands indicates the presence of CS, as indicated by absorption bands around 1645 cm^{-1} (Amide I, C=O, stretching) and 1595 cm^{-1} (N-H, bending of the primary amine groups) [24]. The peaks observed in the 1000–1100 cm^{-1} region include the C-O-C stretching vibrations of the glucosamine rings, which are typical fingerprints of the CS polysaccharide structure. Analysis of SiO_2 Spectrum (Fig. 2B) Siloxane Network (Si-O-Si) The most prominent peak at around 1087 cm^{-1} represents the asymmetric stretching vibrations of Si-O-Si bonds, confirming the formation of the silica network. The band observed at 800 cm^{-1} is characteristic of the symmetric stretching of Si-O-Si bonds. The peak at 462 cm^{-1} is attributed to the O-Si-O bending vibration mode. Silanol Groups (-Si-OH): The wide band centered at 3440 cm^{-1} is correspond to the Si-OH groups stretching vibrations and absorbed moisture on the nanoparticle surface [25, 26]. The FT-IR results confirm the chemical purity and the functional groups of the starting materials (PVA, CS and SiO_2). The presence of hydroxyl groups in both spectra suggests a high potential for hydrogen bonding between the hydrogel matrix and the SiO_2 during the formation of the NCHGs [25, 26]. Overall, the results confirmed successful formation of NCHG.

X-ray diffraction (XRD) Test

The XRD pattern of the NCHGs (Fig. 3B) confirms the integration of SiO_2NPs into the PVA/CS matrix. This structural evolution is an indicator of the strong interaction at the interface between the nanoparticles and the polymeric chains in the NCHG structure.

(Fig. 3A) The XRD pattern of the SiO_2 nanoparticles has a broad peak centred at a 2θ value of around 20–25 degrees, which is characteristic of an amorphous structure [27]. The lack of sharp, intense peaks indicates that the SiO_2 do not have a long-range crystalline order, which is typical for synthetic amorphous SiO_2.

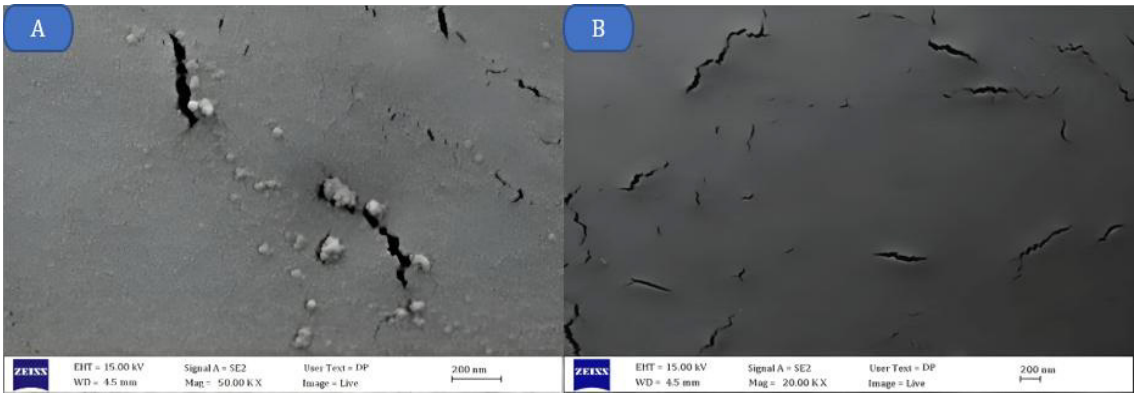

Fig. 4. Image of scanning electron microscopy (SEM) of silica nanoparticles were observed to be distributed within the fractured regions (A), indicating their successful integration and homogeneous dispersion within the hydrogel network of the NCHG. Nanocomposite hydrogel (B), showing a rough and porous surface with visible crack-structures. These cracks enhance water penetration and enhance drug diffusion within the hydrogen matrix confirming its optimal structure for integration.

Analysis of the NCHGs, (Fig. 3B), the sharp peak observed at approximately 2θ=19-20̊ corresponds to the (101) semi-crystalline plane of PVA [20]. The presence of the broad amorphous "hump" alongside the sharper polymer peak suggests that the amorphous nanoparticles are well-dispersed within the hydrogel matrix. For the secondary peaks, the peaks appearing at higher 2θ values (around 40° and 73°) may be attributed to the localized crystalline structure in the polymer-nanoparticle network [5].

Scanning Electron Microscopy (SEM) test:

The SEM image (Fig. 4) reveals a smooth background, which represents the polymer matrix of the hydrogel. Dispersed across this surface are bright, distinct spherical entities representing the SiO_2 nanoparticles. At 20.00 KX magnification with a scale bar of 200 nm, it can be determined that the individual particles are clearly within the nanometric range. This image appears monodisperse, as the dots are evenly distributed and embedded in the background, which is crucial in preventing sudden release of the drug and ensuring controlled release [3].

The bright, small dots observed in the image are clear evidence of the presence of the SiO_2 nanoparticles in the hydrogel. The presence of these bright dots indicates that the drug is possibly adsorbed on the surface of the silica or around these nano-sites, as the dots are evenly distributed, which is crucial for biomedical applications [20].

Swelling evaluation of hydrogel

The ES results for the PVA/CS/SiO_2 NCHG are summarized in the Table 1, clearly indicating the relationship between the structure, as reinforced by the nanoparticles, and the physical response of the polymer material. The NCHG has a Tensile Strength of 3.76 MPa and Young's Modulus of 1.142, indicating that the material is both strong and flexible [22]. The SiO_2 nanoparticles, as reinforcing fillers, restrict the mobility of the PVA/CS chains, enhancing the hardness of the nanocomposite material. The high Elongation at break percentage (338%) also indicates that the material retains good ductility, an important property that allows the material to withstand physiological stresses without failure [7, 22].

The Shore A hardness of 37.79 with a resilience of 33.2% also indicates that the material has viscoelastic properties, suggesting that the nanocomposite material recovers well after deformation, probably as a result of the strong interfacial interactions, as indicated by the strong interfacial hydrogen bonding between the SiO_2 nanoparticles and the PVA/CS matrix, as suggested by the FTIR spectra[21].The NCHG show a well-controlled swelling behaviour in distilled water, with swelling rate gradually decreases over 48 hours, indicating that the SiO_2 nanoparticles, by enhancing the cross-linking density, reduce the volume of the pores, creating a controlled environment for the sustained release of the drug material[1, 3, 19]. The high surface-to-volume

Table 1. Mechanical test of the prepared PVA/CS/ SiO_2/NCHG at dry conditions and after 48 h swelling in distilled water.

Mechanical test	PVA\ CS\SiO_2 NCHG	Swelling in distilled water (48 hr)	Swelling in PBS (48hr)
Tensile strength MPa	3.76	2.61	2.3
Tear resistance MPa	3.17	3.08	2.89
Compression MPa	14.41	8.97	5.87
Hardness shore A	37.79	33.12	30.12
Young modulus MPa	1.142	0.97	0.89
Elongation %	338	354	387
Resilience %	33.2	21.6	19.85

ratio of silica nanoparticles enhances drug adsorption by increasing active sites, improving drug encapsulation efficiency. Modulating PVA concentration in the presence of silica fine-tunes loading capacity and release kinetics for specific therapies. Additionally, silica promotes drug delivery by forming a responsive three-dimensional environment to external stimuli, while ensuring excellent biocompatibility, consistent with prior research findings overall, the developed hydrogel with observed swelling characteristics possesses desirable structural stability, water retention capability, making it is potential for advanced biomedical application including drug delivery system, tissue regeneration and wound dressing [28].

The developed CS/PVA–SiO_2 NCHGs demonstrate significant potential as effective drug delivery systems. Their highly porous, three-dimensional network and strong water absorption capacity, enable efficient drug loading and retention, while the incorporation of SiO_2 enhances structural stability and provides additional binding sites for therapeutic agents. The homogeneous dispersion of SiO_2 within the polymer matrix ensures uniform drug distribution and minimizes burst release [28].

Furthermore, the semi-crystalline nature of the hydrogel, combined with its interconnected morphology, supports controlled and sustained drug release behavior. The presence of functional groups such as –OH and $–NH_2$ facilitates interactions with drug molecules, improving encapsulation efficiency and release modulation. Overall, these properties suggest that the synthesized nanocomposite hydrogels are promising candidates for controlled and targeted drug delivery applications, with tunable performance achieved by adjusting the PVA concentration [29].

Previous studies have showed that CS and PVA hydrogel, when investigated individually, the CS offer excellent biocompatibility and PVA show good mechanical and chemical stability [13, 14]. Consistent with these finding, the present work address the imitation of single component systems such as uncontrolled release behaviour, low drug loading efficiency and insufficient mechanical strength through the development of a hybrid hydrogel[15]. The result indicate that the integration of CS, PVA and SiO_2 yield synergistic nanocomposite system with improved PH-responsive, swelling behaviour and mechanical stability [16].

Mechanical testing was performed for the prepared polyvinyl alcohol (PVA), chitosan (CS), and silica nanoparticles (SiO_2 NPs) NCHG at dry conditions and after 48 h swelling in distilled water and phosphate buffer saline (PBS) to investigate the effect of swelling on the structural integrity and elasticity of hydrogel (Table 1). In acidic conditions there is a decrease in mechanical properties after swelling confirming the pH-responsive behavior of the developed hydrogel. The enhanced swelling and polymer chain relaxation in acidic medium facilitated drug diffusion and support its applications as drug delivery for tumour targeting.

MPa (megapascal).

CONCLUSION

A novel PVA/CS/SiO_2 NCHG was developed for localized drug delivery, with FTIR, XRD and SEM analyses confirming its successful structural formation and porosity. The hydrogel shows high mechanical strength (3.76 MPa) and significant elasticity (338% elongation), along with controlled swelling over 48 hours to ensure sustained drug release. The finding indicates that hydrogel have a good mechanical property therefore could be used in different biomedical applications.

ACKNOWLEDGMENTS

We are sincerely grateful to the University of Babylon and college of pharmacy for support of this work.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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