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Article

Rheological Behavior and Chemical Modification of Fluconazole-Containing Cellulose Derivative Systems

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Abstract: Cellulose diacetate (CDA) solutions prepared in acetone–ethanol and acetone–glycerol solvent systems were investigated to determine their suitability for electrospinning applications. The influence of polymer concentration and solvent composition on rheological behavior was evaluated. An increase in CDA concentration led to a significant rise in viscosity, while the presence of glycerol enhanced intermolecular interactions and solution stability. The optimal rheological properties for fiber formation were observed at polymer concentrations of 7.5–10 wt.%. Surface modification of CDA was performed by alkaline deacetylation to improve hydrophilicity. Fourier-transform infrared spectroscopy (FTIR) confirmed the conversion of acetyl groups into hydroxyl groups, indicating successful deacetylation. The obtained results demonstrate the crucial role of solution rheology and chemical modification in tailoring CDA-based materials for biomedical and drug delivery applications.

Keywords: Cellulose diacetate, rheology, viscosity, deacetylation, FTIR spectroscopy, electrospinning solutions, biomaterials.

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1. Introduction

Fungal infections remain among the most common infectious diseases worldwide and continue to pose a serious threat to public health. The prevalence of opportunistic fungal pathogens, particularly Candida species, has increased significantly during recent decades due to the growing number of immunocompromised patients, prolonged antibiotic therapy, and chronic systemic disorders [1]. Although various antifungal agents are available for clinical use, treatment effectiveness is often limited by inadequate drug concentration at the infection site, insufficient residence time, and the necessity for repeated administration.[2] Consequently, considerable attention has been directed toward the development of advanced drug delivery systems capable of improving therapeutic efficacy while reducing systemic side effects [3].

Fluconazole is one of the most widely prescribed triazole antifungal drugs owing to its broad-spectrum activity, favorable pharmacokinetic profile, and relatively low toxicity. It is extensively used in the treatment of oral, vaginal, and systemic fungal infections caused by Candida species and other pathogenic fungi [4]. Despite its clinical success, conventional dosage forms of fluconazole may exhibit limitations associated with rapid drug elimination and non-specific distribution [5]. These challenges have encouraged researchers to investigate polymer-based carrier systems that can enhance drug retention and provide controlled release characteristics.[6]

Among various polymeric materials, cellulose derivatives have emerged as promising candidates for pharmaceutical and biomedical applications. Their popularity is primarily attributed to their biocompatibility, biodegradability, non-toxicity, availability from renewable resources, and ease of chemical modification [7]. The presence of reactive functional groups enables structural tailoring of cellulose derivatives, allowing the adjustment of physicochemical properties according to specific application requirements.[8] Such characteristics make cellulose-derived materials attractive platforms for the incorporation and delivery of antifungal agents [9].

The performance of polymer-based drug delivery systems is strongly influenced by the properties of the precursor solutions from which they are prepared. In particular, rheological behavior plays a critical role in determining processability, structural stability, and the quality of the resulting materials [10]. Parameters such as viscosity, flow behavior, and intermolecular interactions are affected by polymer concentration, solvent composition, and the presence of active pharmaceutical ingredients [11]. Understanding these relationships is essential for optimizing formulation conditions and ensuring reproducible material characteristics.

Solvent selection is another important factor affecting polymer solution behavior.[12] Different solvent systems can alter chain conformation, molecular interactions, and solution homogeneity, thereby influencing subsequent processing and material formation [13]. The incorporation of additives such as plasticizers may further modify rheological characteristics by affecting hydrogen bonding and molecular mobility within the polymer matrix [14]. Therefore, comprehensive investigation of solution properties is required to establish optimal conditions for the preparation of functional pharmaceutical materials.

In addition to rheological considerations, surface chemistry represents a key parameter governing the interaction of polymeric materials with biological environments [15]. Cellulose derivatives containing acetyl groups generally exhibit hydrophobic behavior, which may restrict wettability and drug–matrix interactions [16]. Chemical modification through deacetylation offers an effective strategy for increasing the density of hydroxyl groups on the polymer surface, thereby enhancing hydrophilicity and potentially improving drug loading and release characteristics [17]. Evaluation of such structural transformations requires reliable analytical techniques capable of identifying functional group changes at the molecular level [18]. Fourier-transform infrared spectroscopy (FTIR) is one of the most widely employed methods for the characterization of polymeric materials. FTIR analysis provides valuable information regarding chemical composition, intermolecular interactions, and structural modifications occurring during processing [19]. In cellulose-derived systems, FTIR is particularly useful for monitoring the conversion of acetyl groups into hydroxyl functionalities and for confirming successful chemical modification [20].

Therefore, the aim of the present study was to investigate the rheological behavior of fluconazole-containing cellulose-derived polymer systems prepared using different solvent compositions and to evaluate structural changes induced by chemical modification through FTIR spectroscopy. Particular attention was devoted to understanding the influence of solvent environment on solution viscosity and the effectiveness of deacetylation in altering surface chemistry. The findings contribute to a deeper understanding of structure–property relationships in cellulose-based pharmaceutical systems and may facilitate the development of improved antifungal drug delivery platforms.

2. Materials and Methods

2.1. Materials

Cellulose diacetate (degree of substitution 2.3, degree of polymerization 500), purchased from Aladdin Chemical Co. (China), was used as the polymer matrix without further purification. Fluconazole (purity ≥99%) was obtained from Xian Tian Guangyuan

Biotech Co., Ltd. (China) and used as the model antifungal agent. Acetone, ethanol, glycerol, sodium hydroxide (NaOH), and distilled water were of analytical grade and used without further purification.

2.2. Preparation of Polymer Solutions

Polymer solutions were prepared by dissolving CDA in mixed solvent systems consisting of acetone/ethanol and acetone/glycerol. Three polymer concentrations (5.0, 7.5, and 10.0 wt.%) were prepared.

Solvent systems were adjusted at volume ratios of 80/20, 85/15, and 90/10 (acetone/co-solvent). The polymer was gradually added into the solvent under continuous magnetic stirring at room temperature (25 ± 2 °C) until homogeneous solutions were obtained. The solutions were further stirred for 4 h and left to equilibrate for 24 h prior to use.

2.3. Fabrication of Drug-Loaded Structures

Fluconazole was incorporated into the prepared polymer systems using ethanol-based solutions at concentrations of 0.1%, 0.5%, and 1.0%. Drug loading was performed by post-treatment adsorption, where the obtained structures were immersed in fluconazole solutions for a defined period to allow drug uptake and surface deposition.

After loading, the samples were dried at ambient conditions until constant weight was achieved.

2.4. Alkaline Surface Treatment

Selected samples were treated with aqueous sodium hydroxide solution to improve surface hydrophilicity. The alkaline treatment facilitated partial deacetylation of CDA, converting acetyl groups into hydroxyl groups. After treatment, samples were thoroughly washed with distilled water until neutral pH and dried at room temperature.

2.5. Fourier Transform Infrared Spectroscopy (FTIR)

Chemical structure and surface modifications were analyzed using Fourier transform infrared spectroscopy (FTIR) (Bruker, Germany). Spectra were recorded in the range of 4000–400 cm^{-1}.

FTIR analysis was used to confirm the presence of characteristic functional groups and to evaluate structural changes associated with alkaline treatment and drug incorporation.

2.6. Rheological Measurements

The rheological behavior of polymer solutions was investigated using an Anton Paar rotational rheometer. Measurements were conducted at 25 °C under controlled shear conditions to evaluate viscosity and flow behavior as a function of polymer concentration and solvent composition.

2.7. Data Analysis

All experiments were performed in triplicate. Results are presented as mean ± standard deviation (SD). Statistical analysis was performed using OriginPro software, and significance was considered at $p < 0.05$.

3. Results and Discussion

The physicochemical properties of polymer solutions play a decisive role in determining the characteristics of the resulting materials. In cellulose diacetate-based systems, the composition of the solvent mixture, polymer concentration, and intermolecular interactions significantly influence solution behavior. These parameters affect the structural organization of polymer chains, solution stability, and processing performance.

The incorporation of co-solvents into acetone-based systems may alter polymer–solvent interactions and consequently modify the rheological properties of the solution.

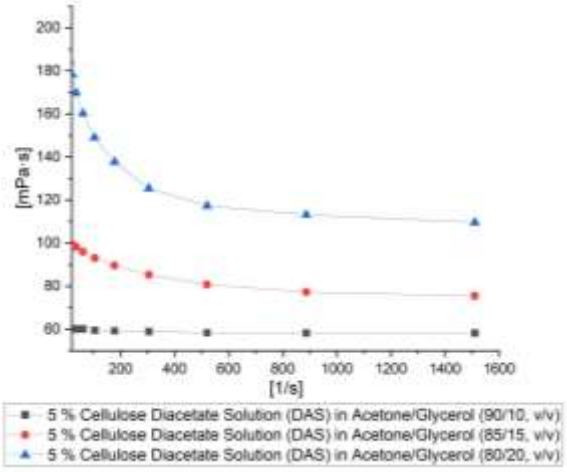
Ethanol and glycerol differ considerably in polarity, hydrogen-bonding ability, and volatility, which can influence the conformation and mobility of cellulose diacetate macromolecules. As a result, changes in solvent composition are expected to affect viscosity and structural properties of the prepared solutions.

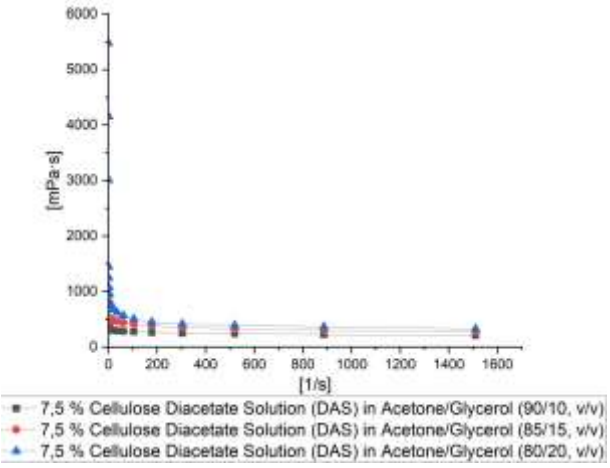
Since the rheological behavior of polymer solutions is closely related to their suitability for further processing and material fabrication, a detailed investigation of viscosity changes associated with polymer concentration and solvent composition was carried out. Particular attention was paid to the influence of acetone–ethanol and acetone–glycerol solvent systems on the structural characteristics of cellulose diacetate solutions.

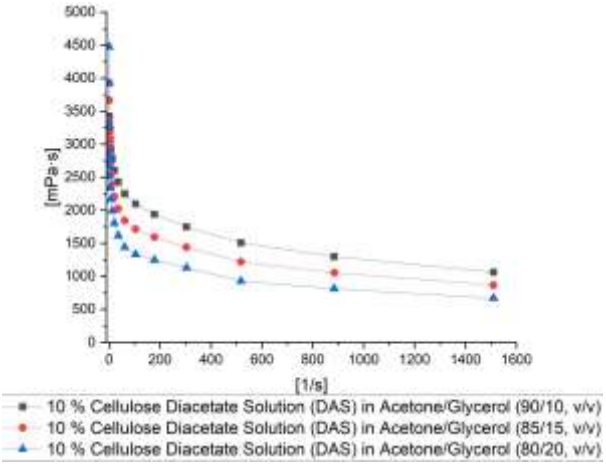
3.1. Rheological Properties of Cellulose Diacetate Solutions

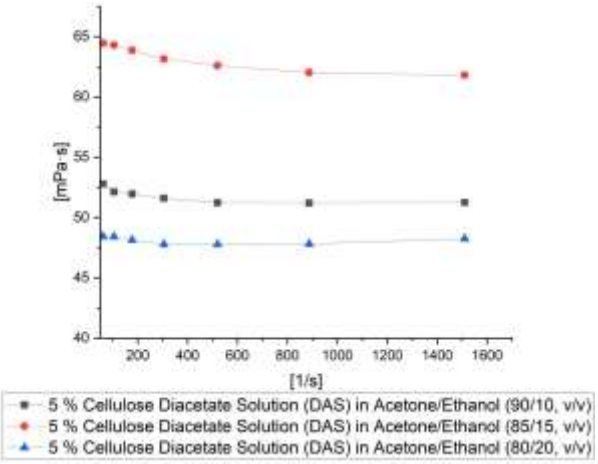
For the fabrication of medical coatings, cellulose diacetate (CDA) solutions of various concentrations were prepared using acetone–ethanol and acetone–glycerol solvent systems, and their rheological properties were investigated.

The Figure 1. structural viscosity of cellulose diacetate solutions is influenced by several factors, including polymer concentration, molecular characteristics, solvent composition, and intermolecular interactions.









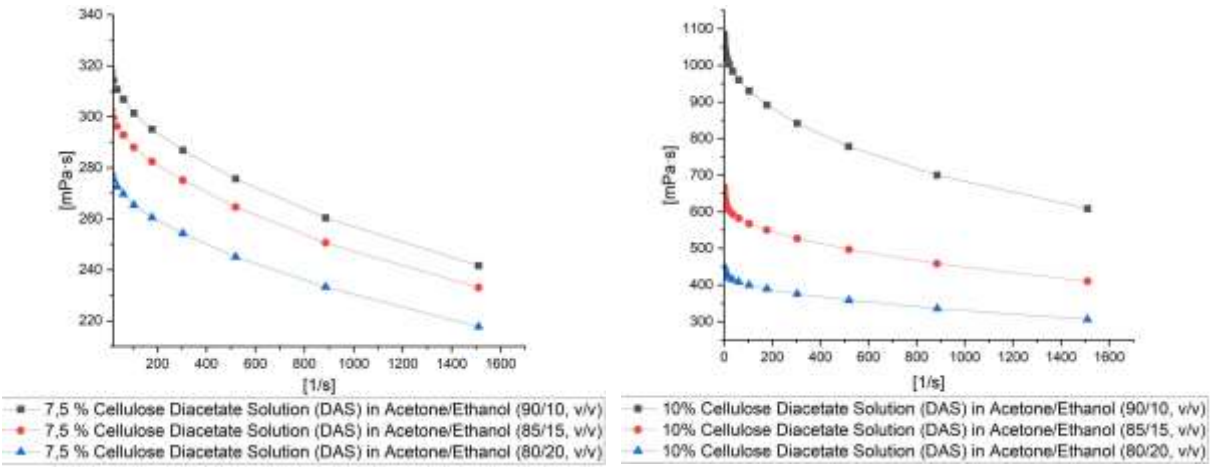


Figure 1. Effect of solvent composition on the viscosity of cellulose diacetate solutions at different polymer concentrations (solvent systems: acetone/ethanol and acetone/glycerol).

Generally, an increase in polymer concentration leads to enhanced chain entanglement and stronger intermolecular interactions, resulting in higher viscosity values. Conversely, dilution reduces the degree of macromolecular association and increases chain mobility, thereby decreasing the structural contribution to viscosity.

The addition of co-solvents such as ethanol and glycerol may further affect the rheological behavior by modifying polymer–solvent interactions. In particular, glycerol is capable of forming hydrogen bonds with cellulose diacetate chains, which may contribute to increased viscosity and altered structural organization within the solution.

Figure 1 presents the rheological behavior of cellulose diacetate solutions prepared at concentrations of 5.0, 7.5, and 10.0 wt.% in acetone-based solvent systems. The investigated formulations contained ethanol or glycerol as co-solvents at concentrations of 10, 15, and 20 vol.%, allowing evaluation of the effect of solvent composition on solution viscosity and flow behavior.

As can be seen, the viscosity of the system increased with increasing cellulose diacetate concentration within the range of 5–10 wt.%. The dilute cellulose diacetate solutions (5 wt.%) exhibited behavior characteristic of Newtonian fluids. This can be explained by the relatively large distances between macromolecules in dilute solutions, resulting in weak intermolecular interactions and a low probability of chain association. In contrast, solutions containing 7.5 wt.% cellulose diacetate and above exhibited non-Newtonian behavior.

The viscosities of solutions containing 10, 15, and 20 wt.% ethanol or glycerol, added to control the solvent evaporation rate, were also investigated. The results showed that the viscosity of glycerol-containing solutions was significantly higher than that of ethanol-containing solutions. Moreover, even 5 wt.% cellulose diacetate solutions containing 15–20 wt.% glycerol exhibited non-Newtonian characteristics. The highly hydrophilic nature of glycerol and the presence of three hydroxyl groups promote the formation of multiple hydrogen bonds with the acetyl and residual hydroxyl groups of cellulose diacetate chains. These interactions restrict chain mobility, maintain the polymer chains in a partially associated state, and increase the internal friction of the solution. In contrast, ethanol molecules contain only one hydroxyl group, limiting the number of hydrogen bonds formed with cellulose diacetate. As a result, intermolecular interactions are weaker and the solution viscosity remains lower. Furthermore, the low volatility of glycerol reduces the overall solvent evaporation rate and enhances the conformational stability of polymer chains within the solution. Consequently, cellulose diacetate solutions containing glycerol exhibit higher internal resistance to flow, which can directly influence the morphology and diameter of nanofibers produced by electrospinning.

The viscosity of cellulose diacetate solutions was also examined as a function of shear rate. It is well known that at low shear stresses, the shear rate is proportional to the applied stress, and under such conditions polymer systems behave similarly to Newtonian fluids. According to the rheological results (Figure 1), increasing cellulose diacetate concentration led to a significant increase in solution viscosity, whereas the dependence of viscosity on shear rate remained largely unchanged.

Based on the rheological analysis, cellulose diacetate solutions containing 7.5–10 wt.% polymer were selected as optimal formulations for the electrospinning process. Using these solutions, the electrospinning conditions for nanofiber fabrication were further investigated. The effects of polymer concentration and solvent composition on key electrospinning parameters, including solution feed rate and applied voltage, were evaluated to determine the optimal conditions for nanofiber formation.

3.2. Deacetylation of Cellulose Diacetate Materials and FTIR Characterization

Cellulose diacetate contains a significant number of acetyl groups, which impart hydrophobic properties to the material. While such characteristics contribute to structural stability, they may limit wettability and reduce interactions with biologically active compounds. For drug delivery applications, increasing the hydrophilicity of the polymer surface is often desirable in order to enhance the incorporation, retention, and release of active agents. Therefore, alkaline treatment was applied to selected cellulose diacetate-based materials to induce deacetylation and generate hydroxyl-rich surfaces.

The deacetylation process involves the hydrolysis of ester bonds in cellulose diacetate, resulting in the conversion of acetyl groups (-$OCOCH_3$) into hydroxyl groups (-OH). The newly formed hydroxyl groups can participate in hydrogen bonding and other intermolecular interactions, thereby improving the affinity of the material toward pharmaceutical compounds and aqueous environments. Such structural modification is expected to increase the potential applicability of cellulose-derived materials in antifungal drug delivery systems.

Following alkaline treatment, slight structural changes were observed in the samples. Swelling of the polymer matrix during treatment, followed by drying, resulted in a moderate increase in the average diameter of the fibrous structures. The obtained diameters remained within the range of approximately 600–800 nm, indicating that the treatment did not lead to significant structural degradation of the material.

The chemical changes associated with deacetylation were investigated using FTIR spectroscopy, and the corresponding spectra are presented in Figure 2. The FTIR spectra of the untreated samples exhibited characteristic absorption bands typical of cellulose diacetate. In particular, the absorption band observed at approximately 1240 cm^{-1} was assigned to the C–O stretching vibration of the ester group. The band located near 1380 cm^{-1} corresponds to the deformation vibrations of methyl groups associated with the acetyl substituents. In addition, a strong absorption band at approximately 1750 cm^{-1} was attributed to the stretching vibration of the ester carbonyl group (C=O), which represents one of the most characteristic features of cellulose diacetate.

Significant changes were observed after alkaline treatment. As shown in Figure 2, samples 1 and 4 displayed intense absorption bands characteristic of cellulose diacetate, indicating the preservation of acetyl functionalities. In contrast, samples 2 and 3 exhibited a pronounced decrease in the intensity of the bands at 1240, 1380, and 1750 cm^{-1}. The reduction or near disappearance of these peaks indicates the cleavage of ester groups and confirms the occurrence of deacetylation.

Simultaneously, an increase in the intensity of the broad hydroxyl absorption region was observed, further supporting the formation of hydroxyl groups on the polymer surface. These results demonstrate that alkaline treatment effectively converted a substantial portion of acetyl groups into hydroxyl functionalities without destroying the

overall structural integrity of the material. The Figure 2. successful deacetylation of cellulose diacetate is expected to improve surface hydrophilicity and create additional active sites for interaction with antifungal compounds, thereby enhancing the functional performance of the developed drug delivery systems.

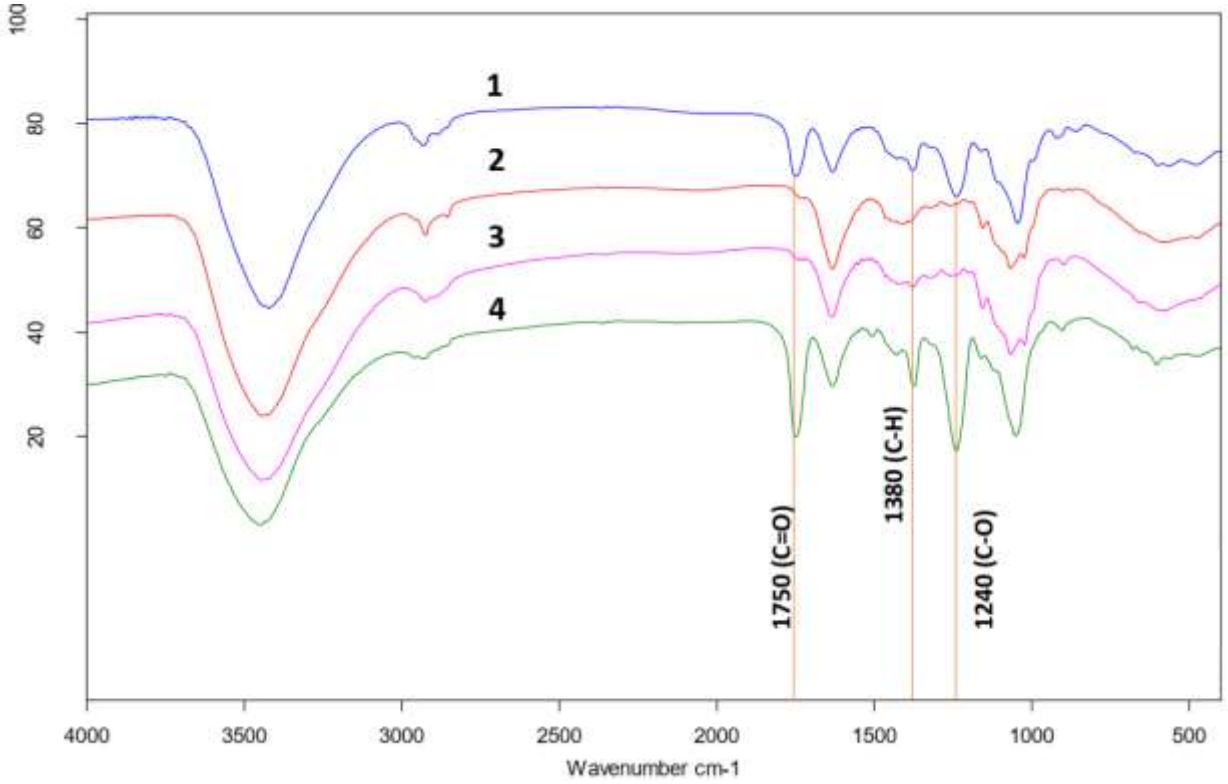


Figure 2. FTIR spectra of cellulose diacetate-based materials before and after deacetylation and fluconazole incorporation: (1) cellulose diacetate fibers; (2) deacetylated cellulose diacetate fibers; (3) deacetylated cellulose diacetate fibers loaded with fluconazole; and (4) cellulose diacetate fibers loaded with fluconazole.

To evaluate the successful incorporation of fluconazole into the cellulose diacetate-based materials, FTIR spectra of the drug-loaded samples were analyzed and compared with those of the untreated materials. Fluconazole possesses several characteristic functional groups that can potentially be identified in the infrared spectra, including hydroxyl, aromatic, triazole, and difluorophenyl moieties.

Pure fluconazole is characterized by broad O–H stretching vibrations in the region of 3200–3150 cm^{-1}, while aromatic and aliphatic C–H stretching vibrations appear in the ranges of 3010–3000 cm^{-1} and 2950–2850 cm^{-1}, respectively. Additional characteristic bands are associated with aromatic ring vibrations, triazole ring vibrations, and C–N stretching modes. A particularly important spectral feature of fluconazole is the absorption band corresponding to C–F stretching vibrations of the difluorophenyl group, which is typically observed in the region of 1020–1000 cm^{-1}.

As shown in Figure 2, most characteristic absorption bands of fluconazole were not clearly distinguishable in the spectra of the drug-loaded samples. This observation can be attributed to the relatively low amount of incorporated drug as well as the overlap of fluconazole absorption bands with the broad and intense absorption signals of the cellulose-based matrix. Similar effects have been reported for polymer–drug systems in which the concentration of the active compound is insufficient to generate prominent spectral features against the background of the polymeric carrier.

Despite this overlap, important evidence of fluconazole incorporation was obtained from samples 2 and 3. In these spectra, a distinct absorption band at approximately 1024 cm^{-1} was observed. This band corresponds to the stretching vibration of the C–F bond in

the difluorophenyl fragment of fluconazole and is considered one of the most characteristic infrared markers of the drug molecule. Since cellulose diacetate does not exhibit a strong absorption band at this specific position, the appearance of the 1024 cm^{-1} peak provides strong evidence for the presence of fluconazole within the modified materials.

The preservation of the characteristic C–F absorption band, together with the absence of significant shifts in the major polymer bands, suggests that fluconazole was incorporated into the cellulose-based matrix without substantial chemical degradation or structural alteration of the drug molecule. These findings indicate that the loading procedure enabled successful incorporation of fluconazole while maintaining the structural integrity of both the polymer carrier and the active pharmaceutical ingredient.

Overall, FTIR analysis confirmed both the successful deacetylation of cellulose diacetate and the incorporation of fluconazole into the modified polymer matrix, demonstrating the suitability of the developed materials for antifungal drug delivery applications.

Conclusion

In this study, cellulose diacetate-based polymer systems containing fluconazole were prepared using acetone–ethanol and acetone–glycerol solvent mixtures, and their rheological and structural properties were investigated. The results demonstrated that both polymer concentration and solvent composition significantly influenced the behavior of the prepared solutions. An increase in cellulose diacetate concentration resulted in higher viscosity due to enhanced intermolecular interactions and macromolecular entanglement, while the incorporation of ethanol or glycerol altered the rheological characteristics through changes in polymer–solvent interactions.

Among the investigated formulations, solutions containing 7.5–10 wt.% cellulose diacetate exhibited rheological properties suitable for the preparation of uniform fibrous materials. The presence of glycerol generally increased solution viscosity compared with ethanol-containing systems, indicating stronger intermolecular interactions and hydrogen-bonding effects within the polymer matrix.

Alkaline treatment successfully induced deacetylation of cellulose diacetate, leading to the conversion of acetyl groups into hydroxyl functionalities. FTIR analysis confirmed this structural transformation through the reduction of characteristic ester-related absorption bands and the increased contribution of hydroxyl groups. The obtained results indicate that the applied modification method effectively enhanced the chemical functionality of the material surface without causing significant structural degradation.

FTIR analysis of fluconazole-loaded samples further demonstrated the successful incorporation of the antifungal agent into the modified polymer matrix. Although most characteristic absorption bands of fluconazole were partially masked by the polymer spectrum, the appearance of the characteristic C–F stretching vibration at approximately 1024 cm^{-1} provided evidence of drug presence within the material.

Overall, the combination of controlled solution formulation and post-treatment modification enabled the development of cellulose diacetate-based materials with tailored rheological behavior and enhanced surface functionality. The obtained findings provide a foundation for the design of advanced cellulose-derived carriers intended for antifungal drug delivery and other biomedical applications.

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