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Study of IR Spectra of Initial Components for Starch-Based Films Modified with Silver Nanoparticles Obtained using Different Reducing Agents

Khodjakulova N.D.^{*1}, Sarimsakov A.A.^{1,2}, Mukhamadiev N.K.¹, Avazova N.¹

1. Samarkand State University named after Sh. Rashidov, Uzbekistan

2. Institute of Chemistry and Physics of Polymers, Uzbekistan

* Correspondence: xodjaqulova_nazokat@samdu.uz

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Abstract: In this work, the spectral characteristics of the initial components used for the preparation of starch-based films modified with silver nanoparticles (AgNPs) were studied by FTIR spectroscopy. The components included two starch samples (Starch-1 and Starch-2), ascorbic acid, and three modifications of citric acid (Citric acid-1, -2, -3). A comparative analysis of the positions, intensities, and widths of the main absorption bands in the regions 900–1500 and 3000–3500 cm⁻¹ was carried out. Characteristic differences due to the chemical structure and the degree of hydration of the samples were revealed. The obtained spectral data can serve as a reference basis for the subsequent identification of structural changes upon introduction of silver nanoparticles into the polymer matrix. It was established that FTIR spectroscopy is an effective express method for preliminary quality control of the initial reagents for the synthesis of biocomposite materials.

Keywords: FTIR Spectroscopy, Starch, Citric Acid, Ascorbic Acid, Silver Nanoparticles, Hydrogen Bonds, Reducing Agents

1. Introduction

In recent years, biocomposite materials based on natural polymers modified with metal nanoparticles have attracted considerable research interest [1-3]. Among such systems, starch-based films containing silver nanoparticles (AgNPs) hold a special place due to their pronounced antibacterial properties and their applications in packaging materials, medical devices, and biosensors [4-6].

Starch is an accessible, biocompatible, and biodegradable polysaccharide capable of acting simultaneously as a reducing agent and stabilizer in the synthesis of silver nanoparticles [1, 2, 7]. The hydroxyl groups and the reducing end of the hemiacetal of starch provide the reduction of Ag⁺ ions to metallic silver, as confirmed by a number of studies [8-10]. To increase the efficiency of synthesis and to control the particle size, additional reducing agents are often used, among which citric acid and ascorbic acid are the most common [11-13]. These organic acids not only participate in the reduction process but can also be incorporated into the polymer matrix, affecting the structure and properties of the final films [14, 15].

Fourier-transform infrared (FTIR) spectroscopy is one of the most informative methods for studying the structure of polymeric materials, allowing the identification of

functional groups, assessment of the nature of intermolecular interactions, and detection of structural changes occurring upon modification [16, 17]. It has been shown in studies [3, 9, 16] that the introduction of silver nanoparticles into the starch matrix leads to characteristic changes in the IR spectra: narrowing and shift of the O–H stretching vibration band ($3200\text{--}3500\text{ cm}^{-1}$), broadening and red shift of the C–O–C bond peaks ($1000\text{--}1150\text{ cm}^{-1}$), as well as a decrease in the intensity of the C–H bond bands ($2850\text{--}2950\text{ cm}^{-1}$). Moreover, the disappearance of peaks at 1384 and 2122 cm^{-1} may serve as a marker of complete reduction of silver ions and oxidation of starch hydroxyl groups [16, 17]. The choice of reducing agent (citric or ascorbic acid) also affects the spectral characteristics, particularly in the region of carbonyl groups ($\approx 1700\text{--}1750\text{ cm}^{-1}$) and in the fingerprint region ($900\text{--}1500\text{ cm}^{-1}$) [11, 19, 20].

The aim of the present work is to perform a systematic analysis of the spectra of the initial components (starch, citric acid, and ascorbic acid) by FTIR spectroscopy in order to create a database of reference data necessary for the subsequent study of films modified with silver nanoparticles synthesized using different reducing agents.

2. Materials and Methods

Objects of study. The objects of study were six commercial samples whose transmission spectra were recorded by FTIR spectroscopy: two starch samples (Starch-1 and Starch-2), ascorbic acid, and three samples of citric acid (Citric acid-1, Citric acid-2, and Citric acid-3), representing different modifications (e.g., anhydrous, monohydrate, or different degrees of crystallinity). In this study, films modified with silver nanoparticles were not analyzed — the work is a preparatory stage for the creation of a spectral database.

Spectral registration. Transmission spectra were recorded in the mid-infrared range ($400\text{--}4000\text{ cm}^{-1}$) on a Spectrum Two FTIR spectrometer (PerkinElmer) with a resolution of 4 cm^{-1} and 32 scans. The samples were prepared as pellets pressed with potassium bromide (KBr) in a mass ratio of 1:100. For each sample, three independent measurements were performed and the spectra were averaged. Baseline correction was performed using the convex hull method with Spectrum 10 software.

Processing of spectral data (smoothing, peak fitting, measurement of maximum positions and half-widths) was carried out using OriginPro 2021 software.

3. Results and Discussion

Analysis of the obtained IR spectra showed that all studied samples have a similar set of characteristic bands, but differ markedly in intensity and width, especially in the regions $900\text{--}1500\text{ cm}^{-1}$ and $3000\text{--}3500\text{ cm}^{-1}$ (Fig. 1).

Table 1 presents the interpretation of the main absorption bands, performed on the basis of literature data [1, 16, 17, 21].

Both starch samples (Starch-1 and Starch-2) exhibit a broad band in the region $3200\text{--}3500\text{ cm}^{-1}$ with maxima at 3295 cm^{-1} (Starch-1) and 3308 cm^{-1} (Starch-2), corresponding to stretching vibrations of O–H groups involved in inter- and intramolecular hydrogen bonds. The band around 2928 cm^{-1} is associated with $\nu(\text{C–H})$ vibrations. In the region $900\text{--}1200\text{ cm}^{-1}$, due to vibrations of C–O and C–O–C glycosidic bonds, characteristic peaks are observed at 1082 cm^{-1} (C–O of the anomeric carbon atom) and 1018 cm^{-1} (symmetric C–O–C vibrations). The spectra of Starch-1 and Starch-2 are similar; however, Starch-2 shows a slightly higher intensity of the band at 1018 cm^{-1} , which may indicate a higher degree of crystallinity or a different amylose/amylopectin content.

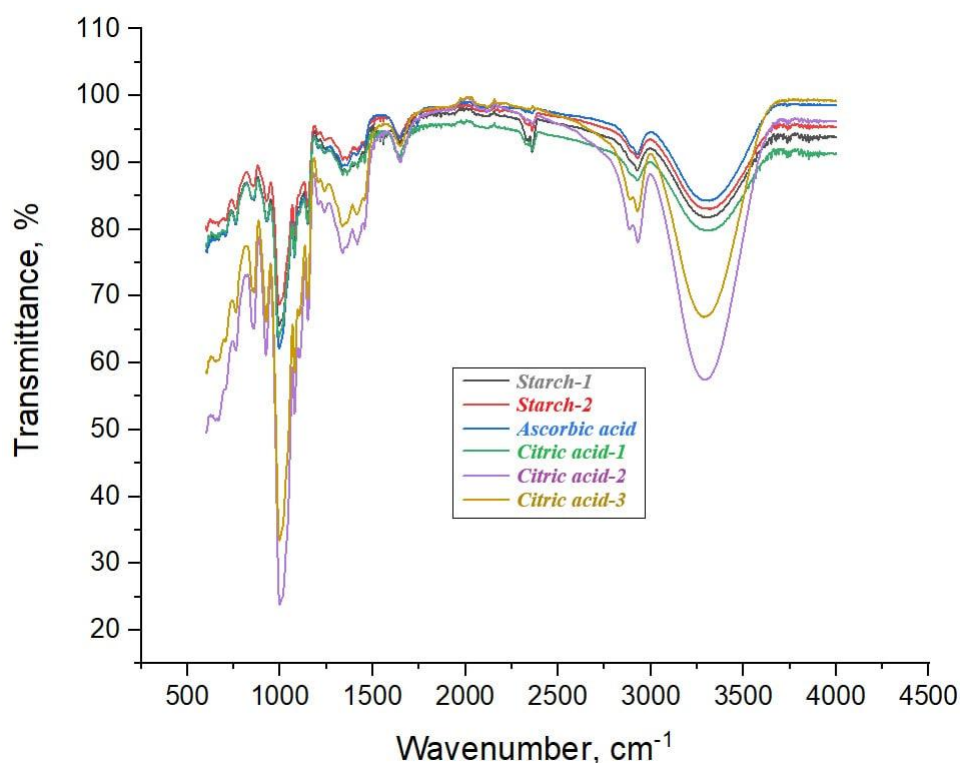


Figure 1. IR spectra of the studied samples

Table 1 . Assignment of the main bands in the IR spectra of the studied samples

Region, cm ⁻¹	Band assignment	Interpretation
3200–3500	$\nu(\text{O-H})$	Stretching vibrations of hydroxyl groups; a broad band indicates an extensive system of hydrogen bonds and contribution of adsorbed/bound water
2850–2950	$\nu(\text{C-H})$	Stretching vibrations of aliphatic CH and CH ₂ groups
≈1700–1750	$\nu(\text{C=O})$	Carbonyl groups of –COOH; particularly characteristic of citric and ascorbic acids
≈1550–1650	$\delta(\text{H-O-H})$, $\nu(\text{C=O})$	Deformation vibrations of water and contribution of carbonyl/conjugated structures
≈1350–1450	$\delta(\text{CH}_2)$, $\delta(\text{O-H})$, $\nu(\text{C-O})$	Deformation vibrations of the organic skeleton
≈1150–1250	$\nu(\text{C-O})$, $\nu(\text{C-O-C})$	Vibrations of ether and alcohol C–O bonds
≈1000–1150	$\nu(\text{C-O})$, $\nu(\text{C-O-C})$	Intense “fingerprint” region of carbohydrates and oxygen-containing functional groups
≈900–1000	C–O–C and ring skeleton vibrations	Characteristic of the polysaccharide structure of starch

The spectrum of ascorbic acid contains a broad O–H band in the region 3200–3500 cm^{-1} with a maximum at 3340 cm^{-1} . The presence of numerous hydroxyl groups gives rise to pronounced hydrogen bonding. Bands at 1750 cm^{-1} (C=O of the lactone ring) and 1650 cm^{-1} (conjugated C=C–C=O system) are characteristic of the ascorbic acid structure. Intense peaks in the 1000–1300 cm^{-1} region are attributed to C–O bond vibrations (1120, 1050, 1020 cm^{-1}).

For the citric acid samples, significant differences in the intensity and position of the bands are observed depending on the modification. Broad absorption in the region 3000–3500 cm^{-1} (maxima at 3250, 3280, and 3220 cm^{-1} for Citric acid-1, -2, and -3, respectively) is related to O–H groups of carboxyl and alcohol groups. In the region ≈ 1700 –1750 cm^{-1} , a characteristic $\nu(\text{C}=\text{O})$ band of carboxyl groups is recorded with maxima at 1725, 1718, and 1730 cm^{-1} for the three samples. The intense structured region 1000–1500 cm^{-1} is mainly due to C–O, C–C, and O–H deformation vibrations.

Particularly noteworthy is the Citric acid-2 sample, which shows the deepest absorption both in the region around 1000 cm^{-1} (peak at 1045 cm^{-1}) and in the broad O–H region (maximum at 3280 cm^{-1} , half-width 420 cm^{-1}). Citric acid-3 occupies an intermediate position, while Citric acid-1 exhibits significantly lower intensity of these bands.

For quantitative evaluation of the differences, the positions of maxima and half-widths of the main bands were measured; the data are summarized in Table 2.

Table 2. Spectral parameters of characteristic bands of the studied samples

Sample	$\nu(\text{O-H})$ max, cm^{-1}	O–H half- width, cm^{-1}	$\nu(\text{C=O})$ max, cm^{-1}	C–O–C peak ($\approx 1080 \text{ cm}^{-1}$), cm^{-1}
Starch-1	3295	385	–	1082
Starch-2	3308	370	–	1081
Ascorbic acid	3340	410	1750	1120
Citric acid-1	3250	450	1725	1045
Citric acid-2	3280	420	1718	1045
Citric acid-3	3220	460	1730	1043

Differences in the intensity and width of the bands may be associated not only with the chemical state of the samples but also with water content, degree of hydration, nature of hydrogen bonds, and differences in crystal structure (e.g., presence of crystallization water in citric acid monohydrate).

The obtained spectral data are fully consistent with literature values for similar compounds [1, 17, 18, 21, 22]. For example, for citric acid, a C=O band at 1715–1730 cm^{-1} is reported in [11], which correlates with our results. For ascorbic acid, the typical C=O range of 1750–1760 cm^{-1} [9] also matches.

It is important to note that the literature [16, 17] describes that upon introduction of AgNPs into the starch matrix, the following changes are observed: a shift of the O–H band to higher frequencies (e.g., from 3240 to 3261 cm^{-1}), broadening of C–O–C peaks accompanied by a red shift (≈ 5 –10 cm^{-1}), a decrease in the intensity of the C–H peak, and disappearance of peaks at 1384 and 2122 cm^{-1} . Our data on the initial components provide reference points for detecting such changes in future experiments with modified films.

4. Conclusion

The FTIR spectroscopic study of the initial components (starch, citric acid, and ascorbic acid) showed that all samples contain characteristic oxygen-containing functional

groups (O–H, C–O, C–O–C, C=O). The following reference spectral parameters have been established: for starch — O–H maximum at $\sim 3295\text{--}3308\text{ cm}^{-1}$, C–O–C peaks at 1082 and 1018 cm^{-1} ; for citric acid - C=O at $1718\text{--}1730\text{ cm}^{-1}$ depending on the modification; for ascorbic acid - C=O at 1750 cm^{-1} and O–H at 3340 cm^{-1} .

Differences between the samples were revealed, which are due not only to the chemical nature but also to the degree of hydration, crystallinity, and character of hydrogen bonds. These differences are most pronounced in the regions $900\text{--}1500$ and $3000\text{--}3500\text{ cm}^{-1}$, while the positions of the main bands remain unchanged, indicating the preservation of the chemical backbone.

The created spectral database can serve as a reliable basis for the subsequent identification of structural changes upon introduction of silver nanoparticles into the starch matrix. FTIR spectroscopy has proven itself as an effective express method for incoming quality control of initial reagents for the synthesis of biocomposites.

For an in-depth study of the effect of reducing agents on the structure of the final films, additional experiments using UV–Vis spectroscopy, X-ray diffraction analysis, transmission electron microscopy, and thermal analysis are planned.

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