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MOLECULAR SPECTROSCOPIC ANALYSIS OF THE FUNCTIONAL COMPOSITION OF SILK FIBRON

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Abstract
X-ray diffraction analysis of the samples showed that in the range of diffraction angles considered, crystalline reflexes were observed at 2θ=13.09°, 18.63°, 21.21°, 25.33° and 30.39°, which are characteristic of fibroin. The most intense maximum crystallographic reflex, characterizing the distance between planes with Muller indices (010), was observed at the diffraction angle 2θ=21.21°, but at the same time, the crystallographic reflex characteristic of (10-1) planes was observed at the angle 2θ=18.63°. X-ray phase analysis revealed that the fibroin samples were found to have a monoclinic crystal syngony with elementary crystal lattice parameters of a=7.057 Å, b=4.212 Å, c=6.114 Å, α=γ=90.00°, and β = 99.20°.

Keywords: Silk fibroin, x-ray, hydrolysis, micro and nano particles, spectrum.

Introduction

Recently, great attention has been paid to the study of biopolymers in nanostructures, especially to the formation of functional-active micro- and nanoparticles and composite materials based on them [1-4]. In this regard, obtaining bioactive surface-active polymer materials with various functions, determining their structures and properties, and the possibilities of their practical application are considered a topical issue in polymer physics [5,6].

Results and Discussion

X-ray diffraction analysis of Bombyx mori silk fibroin obtained by hydrolysis revealed that within the range of diffraction angles considered, the diffraction angles 2θ=13.09°, 18.63°, 21.21°, 25.33°, 30.39°, which correspond to the crystalline reflections of fibroin, were shifted and the values 2θ=11.10°, 19.25°, 21.42°, 24.59°, 29.50° were observed.

Table 1. Diffractogram results of the fibroin sample

№	Bragg angle2θ, °	Inter-plane distance *d*, Å	Peak intensity half-width FWHM, °	Crystallite sizes, Å	Muller indices, *hkl*
1	13.09	6.76	5.00	16.9	001
2	18.63	4.76	4.99	16.9	10-1
3	21.21	4.18	4.51	18.7	010
4	25.33	3.51	6.50	13.1	110
5	30.39	2.93	7.13	12.05	20-1

X-ray phase analysis showed that the Bombyx mori silk fibroin samples obtained by hydrolysis had an orthorhombic crystal structure with elementary crystal lattice parameters a=7.487 Å, b=8.446 Å, c=4.732 Å, α=β=γ=90.00°.

X-ray diffraction analysis of Bombyx mori silk fibroin obtained by hydrolysis under harsh conditions showed that crystalline reflections were observed at 2θ=10.17°, 14.30°, 15.96°, 19.60°, 21.34°, 25.10° and 29.49°, which are characteristic of fibroin crystals, within the range of diffraction angles considered [7,8].

Table 2. Diffractogram results of a sample of silk fibroin with an hydroxide modulus of 1:6, hydrolysis time of 120 minutes, and temperature of 180 °C

№	Bragg angle2θ, º	Inter-plane distance *d*, Å	Peak intensity half-width FWHM, º	Crystallite sizes, Å	Muller indices, *hkl*
1	11.10	8.00	3.40	25.0	100
2	19.25	4.61	4.40	19.1	001
3	21.42	4.14	3.10	27.4	020
4	24.59	3.62	3.80	22.0	210
5	29.50	3.02	7.00	12.3	201

The most intense maximum crystallographic reflection, characterizing the distance between planes with a Muller index of (100), was observed at a diffraction angle of 2θ=19.0° (Fig. 1).

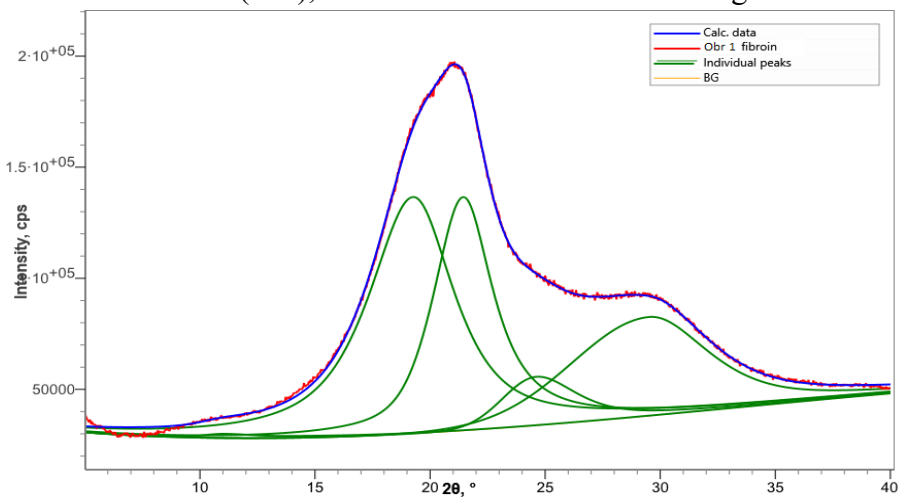

Figure 1. Diffractogram of silk fibroin samples obtained at an hydroxide modulus of 1:6, hydrolysis time of 120 minutes, and temperature of 180 °C

X-ray diffraction analysis of Bombyx mori silk fibroin obtained by hydrolysis under harsh conditions showed that in the range of diffraction angles considered, crystalline reflexes were observed at 2θ=10.17°, 14.30°, 15.96°, 19.60°, 21.34°, 25.10° and 29.49°, which are characteristic of fibroin crystals (Fig. 2).

The most intense maximum crystallographic reflex, characterizing the distance between planes with a Muller index of (100), was observed at a diffraction angle of 2θ=19.0°. X-ray phase analysis revealed that the fibroin samples were found to have a tetragonal crystal structure with

elementary crystal lattice parameters a=4.681 Å, b=4.681 Å, c=16.767 Å, and α=β=γ=90.00° 99.20°.

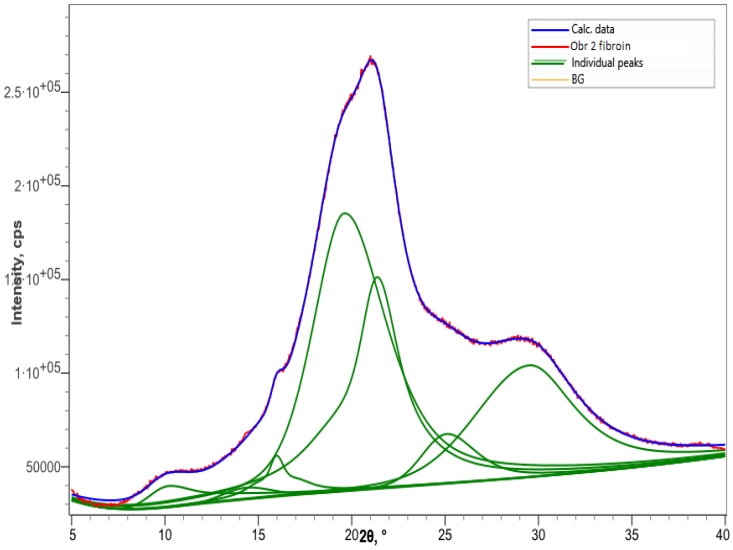

Figure 2. Diffractogram of silk fibroin samples obtained at an hydroxide modulus of 1:6, hydrolysis time of 150 minutes, and temperature of 180 °C

Table 3. Diffractogram results of a sample of silk fibroin with an hydroxide modulus of 1:6, hydrolysis time of 150 minutes, and temperature of 180 °C

№	Bragg angle2θ, º	Inter-plane distance *d*, Å	Peak intensity half-width FWHM, º	Crystallite sizes, Å	Muller indices, *hkl*
1	10.17	8.69	3.30	25.00	002
2	14.30	6.20	3.10	27.00	102
3	15.96	5.54	1.33	63.00	003
4	19.60	4.52	4.70	18.00	100
5	21.34	4.16	3.00	28.00	004
6	25.10	3.55	3.50	25.00	103
7	29.49	3.02	6.27	13.68	112

The conducted studies showed that as a result of hydrolysis, an increase in the degree of crystallization of the obtained fibroin samples was observed.

Increasing the duration of the hydrolysis process leads to an increase in the degree of crystallization of the fibroin samples, including when the hydroxide module is 1:6, the hydrolysis time is 120 minutes, and the temperature is 180 °C, the degree of crystallization of the fibroin is 58%, and when the hydroxide module is 1:6, the hydrolysis time is 50 minutes, and the temperature is 180 °C, the degree of crystallization of the fibroin is 62%.

Conclusion

Increasing the duration of the hydrolysis process leads to an increase in the degree of crystallization of fibroin samples, including when the hydroxide modulus is 1:6, the hydrolysis

time is 120 minutes, and the temperature is 180 °C, the degree of crystallization of fibroin is 58%, and when the hydroxide modulus is 1:6, the hydrolysis time is 50 minutes, and the temperature is 180 °C, the degree of crystallization of fibroin is 62%.

Diffractograms of silk fibroin micro- and nanoparticles were obtained, and an increase in the degree of crystallization was observed in the tested samples depending on the hydrolysis conditions.

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