

<https://doi.org/10.29013/AJT-23-3.4-53-56>

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ON THE MODIFICATION OF CELLULOSE ACETATE WITH PIPERIDINE

Abstract. In this article, the process of modifying cellulose acetate with piperidine in an organic solvent medium (acetone, dimethylformamide) at a temperature of 20–50 °C was studied. The structure and thermal properties of the modified sample were studied by infrared spectroscopy and thermogravimetry.

Keywords: cellulose acetate, cellulose diacetate, piperidine, modification, thermogravimetry.

Introduction

Synthetic membranes acting as a selective barrier are widely used in seawater desalination, separation of dispersed systems and chemicals, for the isolation of rare earth elements, in blood hemodialysis and other areas. The current ideas about the mechanism of transport of molecules and ions through diffusion membranes make it possible in some cases to predict which chemical structure and supramolecular structure of the polymer will be optimal for solving the problems posed. Achieving the required characteristics of membranes is possible by modifying them or synthesizing new polymers. However, it should be taken into account that the synthesis of new polymers is a laborious and expensive process. The modification is used to increase the selectivity and permeability of membranes, or to impart specific properties to them [1–2].

Cellulose acetate is one of the most important cellulose ethers. Depending on the processing method, cellulose acetate can be used for various syntheses (for example, for films, membranes, or fibers) [3]. The review analyzes the methods of chemical modification of cellulose acetates and the prospects

for creating composite materials based on the modified polymer [4].

Cellulose acetate is widely used in the production of filtration membranes, especially in the food, pharmaceutical and medical industries [5]. The most commonly used membrane polymer is secondary cellulose acetate, cellulose diacetate (DAC). Its macromolecules consist of very rigid and dimensionally stable supramolecules.

The purpose of this work is to study the modification of cellulose acetate with a heterocyclic compound – piperidine, to determine the structure and properties of the synthesized products.

Experimental part

Acetylcellulose – white amorphous mass; density 1300 kg/m³. At 230 °C it starts to decompose. Let's dissolve in organic solvents dimethylformamide and acetone. Acetone (dimethyl ketone) is a colorless volatile liquid with a characteristic odor, mixes well with water, density – 792 kg / m³; melting point – 94 °C, boiling point 56.2 °C. N, N-dimethylformamide colorless liquid. Boiling point 153 °C; dissolves well in alcohol. Piperidine (hexahydropyridine), C₅H₁₁N – colorless liquid with a pungent

odor, purified by distillation before use; molar mass 85.15 g/mol, density 0.862 g/cm³; dynamic viscosity 1.573 Pa*s; melting point 8 °C; boiling point 106.17 °C, mixed in any ratio with water, alcohol, ether [6–7].

Method for modifying cellulose acetate with piperidine. The required amount of acetylcellulose was placed in a conical flask and dimethylformamide was poured, and the conical flask was placed on a magnetic stirrer. After dissolution of cellulose acetate, piperidine was added in portions to the conical flask. The reaction proceeds at a temperature of 40–50 °C for 4 hours. The films were synthesized by known methods from polymer solutions [8]. For this purpose, polymeric dimethylformamide solutions were

prepared in advance in various percentages. Dimethylformamide was used to dissolve cellulose acetate. The completeness of its dissolution was determined by the absence of swollen polymer particles on the glass walls.

Obtained results and their analysis

The modification process of cellulose acetate (AC) with piperidine (Pp) was carried out at a temperature of 50 °C in an organic solvent medium. The chemical composition and structure of the resulting polymers were confirmed by elemental analysis, IR spectroscopy, thermogravimetric (TGA) and differential thermogravimetric analysis (DTA).

The IR spectra of polymers based on AS and PP are shown in (Figs. 1.2).

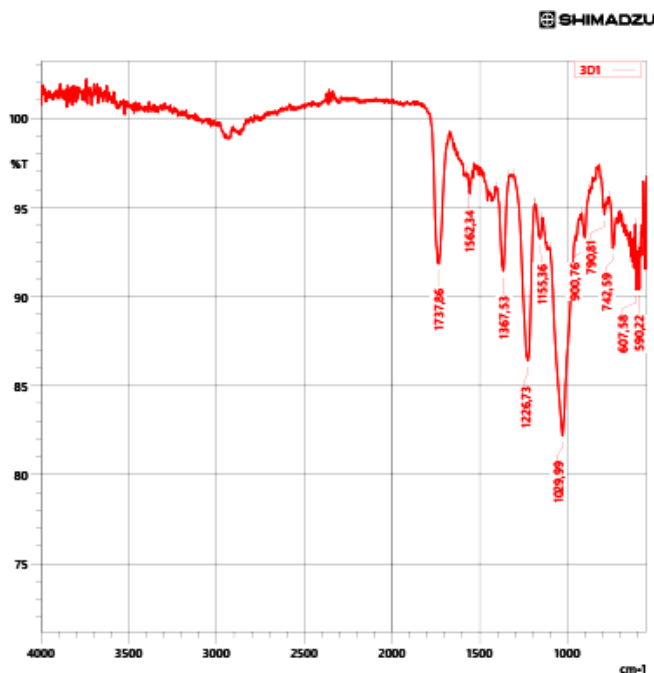


Figure 1. IR spectrum of AC films obtained on the basis of AC and PP

The results of IR spectra show that in the region of 1562.34 cm⁻¹ there is a line of symmetric stretching vibrations of piperidine [8–9].

Based on the experiment and from the results and IR spectroscopic analysis, the reaction between cellulose acetate and piperidine can be expressed as follows:

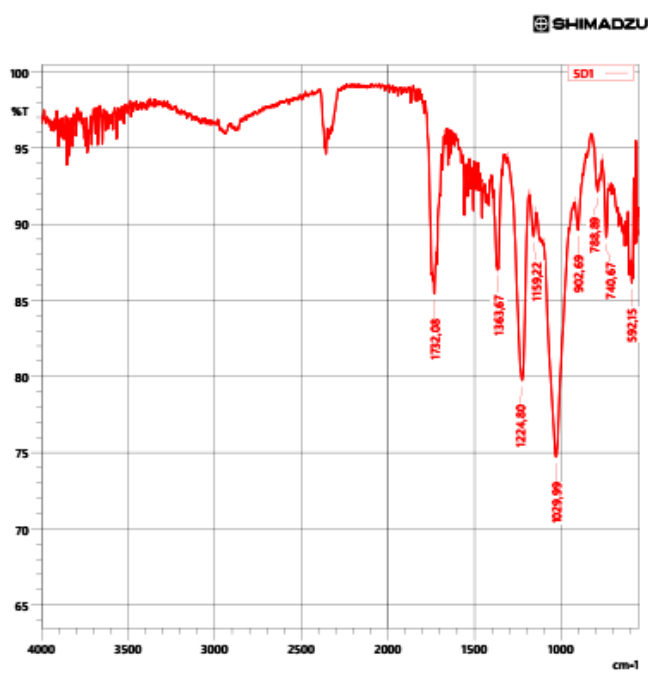


Figure 2. IR spectrum of films

A thermogravimetric study of piperidine with modified samples of cellulose acetate was also carried out (Fig. 3). The results of the analysis of samples were studied on the basis of thermogravimetric derivatogram (TGA) and differential thermogravimetric analysis (DTA). In this case, three endothermic effects were revealed at temperatures of 25.54 °C and 601.34 °C.

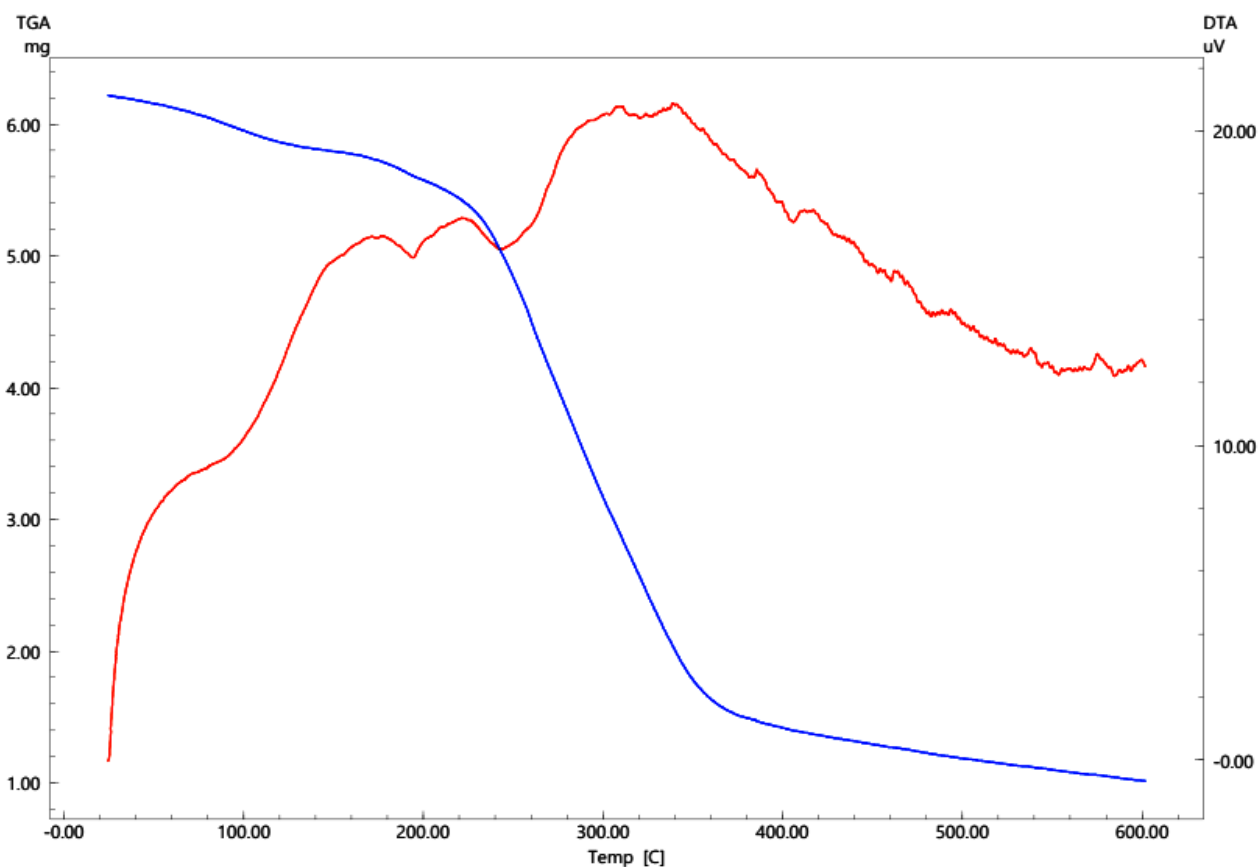
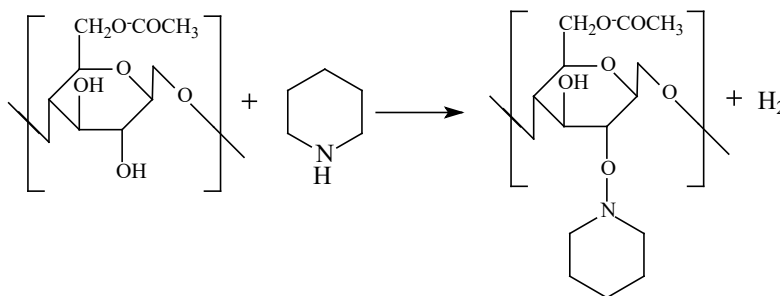


Figure 3. Thermogravimetric derivatogram (TGA) and differential thermogravimetric analysis (DTA) of films based on cellulose acetate and piperidine

A sample weighing 6.217 mg was poured into an open aluminum crucible with a fire resistance of 600 °C, and starting at 20 °C, the temperature was gradually increased to the final one. Analysis of the curve showed that the TGA curve in was formed as a result of intense weight loss within the temperature range: 1-interval of weight loss corresponds to 25.54–162.68 °C, 2-interval of weight loss corresponds to 162.68–378.51 °C, 3-interval of weight loss corresponds to 378.51–601.34 °C. The analysis shows that in the 1st interval the weight loss is

0.451 mg or 7.254%, in the 2nd interval the weight loss is 4.270 mg or 68.683%, and in the 3rd interval the weight loss is 0.483 mg or 7.769% where an intensive decomposition process takes place. The results of the analysis showed that at temperatures of 162.68–378.51 °C, the weight loss is 4.270 mg, or 68.683%. Analysis of the results of thermal decomposition of the substance at different temperatures is given in (Table 1).

Table 1. – Analysis of the results of TGA and DTA curves of modified cellulose acetate

Temperature °C	Lost mass (mg) (relative to 6.217mg)	Lost mass (%)	The amount of energy expended ($\mu V \times s/mg$)	Elapsed time (min)	dw (mg)	dw/dt (mg/min)
50 °C	0.063375	1.02	1.277	3.22	6.153	0.0197
100 °C	0.269324	4.332	1.718	8.42	5.947	0.032
200 °C	0.644124	10.36	2.956	18.55	5.573	0.0347
300 °C	3.050525	49.067	6.475	28.62	3.166	0.106
400 °C	4.803924	77.27	12.52	38.8	1.413	0.124
500 °C	5.033423	80.96	11.7	49	1.183	0.103
600 °C	5.203622	83.7	12.575	59.25	1.013	0.878

Thus, the structure of cellulose acetate modified with piperidine was determined by IR spectroscopy and thermal parameters by thermogravimetry.

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