

# Getting Graft Cellulose Copolymers and Acrylic Monomers

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**Abstract:** Graft copolymers of acrylic monomers with cotton cellulose were obtained. The dependence of the degree and efficiency of grafting of acrylic acid and methyl methacrylate to cellulose on the concentration of monomer and initiator was investigated. Pre-adsorption of the initiator in the macromolecules of cellulose leads to an increase in the efficiency of the grafting. The efficiency of grafting is higher in those systems in which the initiator used is insoluble in the monomer solvent. Absorption of cellulose with an aqueous solution of the initiator - potassium persulfate, followed by removal of water was done. The advantage of using a water-soluble initiator is that during subsequent processing with a solution of monomer in an organic solvent, the desorption of the active centers does not occur. An increase in the concentration of the initiator leads to an increase in the degree of grafting, a slight increase in the efficiency of the grafting, a significant decrease in the degree of polymerization and the molecular weight of the graft chains. In a heterogeneous process, an increase in the efficiency of grafting with an increase in the concentration of the initiator is promoted by the additional adsorption interaction of the initiator molecules with the surface of cellulose. With an increase in the concentration of monomers, the overall degree of conversion slightly increases, the efficiency of grafting slightly decreases, the degree of grafting and the molecular weight of the graft chains increase significantly. The mechanism of graft copolymerization was investigated by comparative analysis of the IR and PMR spectra of cellulose, potassium persulfate, acrylic monomers and products of their interaction. Due to the results of spectroscopic studies, a scheme of graft copolymerization reactions has been proposed. The active centers of graft copolymerization are formed as a result of the reductive interaction of potassium persulfate, water and cellulose macromolecules.

**Keywords :** Cellulose, graft copolymerization, acrylic monomer, initiator.

## I. INTRODUCTION

Grafting acrylic and vinyl monomers to natural polymers is a tool for modifying their properties. Graft radical copolymerization of monomers to cellulose begins with the interaction of the initiator with the cellulose macromolecule

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[1]. Graft copolymers of cotton cellulose with butylmethacrylate were obtained[2]. Pretreatment of cellulose, for example with amines, leads to an increase in the degree of grafting of acrylic and vinyl monomers[3]. Hybrid materials[4] and filter paper[5] based on graft copolymers of cellulose were obtained. Methyl methacrylate is efficiently grafted on cellulose[6] both in homogeneous conditions[7], [8] and in heterogeneous conditions[9]. Grafting of N, N-dimethylacrylamide[10], methacrylic monomers[11], methyl methacrylate[12] to natural silk fibers is carried out according to the same laws as for cellulose. A very important and crucial stage of graft copolymerization is the formation of an active graft site. Potassium[13] or ammonium persulfates[14], hydrogen peroxide[15], tetravalent cerium ions[16], [17], benzoyl peroxide[18], trivalent cobalt ion[19], benzophenone as a photoinitiator[20] and other redox systems may serve as initiators of grafted copolymerization. Under relatively identical reaction conditions, cerium catalysts produce a significantly larger amount of graft copolymer than the method of using divalent ferrous ions. However, the possibility of the formation of a significant amount of a homopolymer is not excluded in all cases, the removal of which from the surface of the fibrous material presents considerable difficulties due to the high consumption of solvents, although these shortcomings are considered to be eliminated. Controversial questions remain about the mechanism of interaction of cellulose with the initiator and monomers, the reaction of growth, breakage, length and molecular weight of graft chains. High vaccination efficiency can be achieved in the case of preliminary application of the initiator by physical adsorption. We conduct systematic studies on the synthesis and properties of graft copolymers of functionally active monomers with collagen[21], natural silk[22],[23], cellulose[24]. Based on graft cellulose copolymers, reinforced[25] and fireproof[26] textile materials were obtained.

The purpose of this study is to increase the efficiency of grafting acrylic monomers to cellulose by preliminary physical adsorption of the initiator and to study the mechanism of graft copolymerization processes.

## II. EXPERIMENTAL PART

Cotton cellulose, polymerization initiator - potassium persulfate (PP), monomers - acrylic acid (AA), methyl methacrylate (MMA), acrylonitrile (AN), and solvents were used in the work. Pulp was obtained and cleaned from a cotton lint in an industrial plant. The monomers and initiator are pre-cleaned in the laboratory using standard methods.



For the graft copolymerization, preliminary physical adsorption of  $PPK_2S_2O_8$  to cellulose was carried out. To do this, a portion of the cellulose was loaded into a glass, poured an aqueous solution of the initiator at the 1: 5 module, closing the lid, kept at room temperature for 1-2 hours. The pulp was removed from the glass, dried at room temperature to constant weight. The synthesis of graft copolymers was carried out in a flask equipped with a stirrer, thermometer and reflux condenser in a stream of nitrogen. Monomer, cellulose with the initiator and the solvent were loaded into the flask, the flask was immersed in a thermostat with a certain temperature. Synthesis of copolymers was carried out with constant stirring of a heterogeneous reaction mixture. The synthesis of AAgraft copolymers carried out in water, MMA - in dioxane, and AN - in dimethylformamide. Upon reaching a certain time, the reaction mixture was removed from the thermostat, cooled to room temperature. Then the reaction mixture was poured into a glass with a solvent of a synthetic polymer, where the graft copolymer does not dissolve, the residual monomer and the polymer unrelated to cellulose remain in solution. Extracting from the mixture, the graft copolymer was purified by the method extraction. The resulting copolymer was placed in a desiccator connected to a vacuum pump and dried to constant weight.

The IR spectra of cellulose, the product of interaction of cellulose with PP, monomers and synthesized copolymers, were recorded on a Parker–Elmer System 2000 FT–IR spectrometer in the wavelength range of 400–4000  $cm^{-1}$ . NMR  $^1H$  (PMR) spectra were taken on a UNITY–400+“Varian” nuclear magnetic resonance spectrometer at an operating frequency of 400 MHz, the solvent was trifluoroamine (TFA).

III. RESULTS AND THEIR DISCUSSION

One of the methods of graft copolymerization under the action of radical initiators is the application of the initiator on the surfaces of solids by preliminary physical adsorption from solution. The efficiency of grafting is higher in those systems in which the initiator used is insoluble in the monomer solvent. For this purpose, cellulose was absorbed by an aqueous initiator solution - PP, followed by removal of water. The advantage of using a water-soluble initiator is that during subsequent processing with a monomer solution in an organic solvent, the desorption of the active centers does not occur due to non-dissolution with an organic solvent. An increase in the initiator concentration leads to an increase in the degree of grafting, a slight increase in the efficiency of grafting, a significant decrease in the degree of polymerization and the molecular weight of the graft chains (table 1).

Table 1

The dependence of the degree and efficiency of grafting of monomers to cellulose and the average molecular weight of graft chains on the concentration of PP. T = 60°C, time - 4 hours

Monomer and its concentration, mole·l <sup>-1</sup>	Concentration of PP, mole·l <sup>-1</sup> · 10 <sup>2</sup>	The degree of conversion, %	The degree of grafting, ng.%	Grafting efficiency, %	The average degree of polymerization	Average MM of grafted chains
AA, 1,39	0,37	52,4	13,4	25,5	98	7000
	0,74	75,3	18,7	24,8	71	5100
	1,11	88,6	23,0	26,0	55	4000
MMA, 1,0	0,37	49,8	8,8	17,7	67	6700
	0,74	69,5	15,3	22,0	47	4700
	1,11	83,7	21,4	25,6	38	3800

In a heterogeneous process, an increase in the efficiency of grafting with an increase in the initiator concentration is promoted by the additional adsorption interaction of the initiator molecules with the surface of cellulose. As expected, the monomer concentration does not affect the degree of conversion and the efficiency of grafting, but it significantly affects the molecular weight of the graft chains (table 2).

Table 2

The dependence of the degree and efficiency of monomers grafting to cellulose and the average molecular weight of graft chains on the concentration of monomers. [PP] = 0.74 · 10<sup>-2</sup> mole·l<sup>-1</sup>, T = 60°C, time - 4 hours

Monomer	The monomer concentration, mole·l <sup>-1</sup>	The degree of conversion, %	The degree of grafting, ng.%	Grafting efficiency, %	The average degree of polymerization	Average MM of grafted chains
AA	0,69	76,2	10,1	26,5	36	2600
	1,39	75,3	18,7	24,8	71	5100
	2,08	78,7	24,5	20,8	112	8100
MMA	0,5	67,4	9,2	27,3	23	2300
	1,0	69,5	15,3	22,0	47	4700
	1,5	68,9	22,7	21,9	70	7000

With an increase in the concentration of monomers, the overall degree of conversion slightly increases, the efficiency of grafting decreases slightly, the degree of grafting, the degree of polymerization and the molecular weight of the graft chains increase significantly. Apparently, the overall degree of conversion depends mainly on the concentration of the initiator, and not the monomer. When the initiator concentration is the same, the number of active initiation centers of the graft copolymerization does not change, and as the monomer concentration increases, the number of molecules that join each active center increases. Naturally, this leads to a proportional increase in the degree of polymerization and the molecular weight of the graft chains.

In order to clarify the mechanism of formation of active centers, we performed spectroscopic studies. The IR and PMR spectra of cellulose, PP, AA, MMA, AN and products of their interaction were analyzed.

In the IR spectrum of cellulose, the characteristic absorption bands of bonds in the macromolecule were detected (table 3). The spectra contain a large number of sharp bands, which is associated with the presence of large areas of high molecular ordering.

Table 3

Characteristic absorption bands in the IR spectrum of cotton cellulose

Absorption band, $cm^{-1}$	Type of vibrations	Band intensity	Bonds and groups of atoms
616	$\delta$	average	C-O-C
665		average	pyranring
897	$\nu$ (symmetrical)	weak	COC
1029	$\delta$	average	CH
1059	$\nu$ (asymmetrical)	average	COC
1115	$\nu$ (asymmetrical)	average	pyranring
1164	$\delta$ (symmetrical)	average	CH
1318	$\delta$ (asymmetrical)	average	CH
1337	$\delta$ (symmetrical)	average	OH
1373	$\delta$ (asymmetrical)	average	OH
1431	$\delta$	average	CH <sub>2</sub>
1641		average	adsorbed water
2899	$\nu$ (symmetrical)	average	CH
2941	$\nu$ (asymmetrical)	weak	CH
3449	$\nu$	wide, strong	OH



Now we analyze the spectra of monomers. In all monomers studied, the characteristic absorption band of the stretching vibrations of double C=C bonds in the region of 1620–1660 cm<sup>-1</sup> is detected. For AA, MMA, ν (C=O) was detected in the frequency range of 1703-1732 cm<sup>-1</sup>, ν (C≡N) AN in the region of 2240 cm<sup>-1</sup>. Valence and deformation vibrations of the vinyl group –CH=CH<sub>2</sub> monomers have a strong signal intensity. These bands are approximately in the same region as the absorption bands of saturated methylene groups. In this case, it is not entirely clear which of the absorption bands belongs to the =CH– group or =CH<sub>2</sub> group. In MMA molecules, the absorption bands of these groups are apparently aligned with the absorption bands of the limit CH, CH<sub>2</sub> and CH<sub>3</sub> groups.

In the PP spectra, the strong absorption band at 1059 cm<sup>-1</sup> refers to the symmetric stretching vibrations S=O. The group of bands in the 2136–2570 cm<sup>-1</sup> areas probably belong to cumulated double bonds O=S=O, and in the 558–690 cm<sup>-1</sup> areas – to the ion. The doublet at 1262-1295 cm<sup>-1</sup> most likely appears as a result of vibrations of the bonds of persulfate (table 4).

**Table 4**

Characteristic absorption bands in the IR spectra of PP

Absorptionband, cm <sup>-1</sup>	Typeofvibrations	Bandintensity	Bonds and groups of atoms
558		average	SO <sub>2</sub> <sup>2-</sup>
690		average	SO <sub>2</sub> <sup>2-</sup>
1059	ν (symmetrical)	strong	S=O
1262	ν (symmetrical)	strong	O–O
1295	ν (asymmetrical)	strong	O–O
2136	ν	average	O=S=O
2570	ν	average	O=S=O

To clarify the initiation mechanism, infrared spectroscopic studies of the interaction of cellulose with PP were carried out under the same conditions as the starting materials. At the same time, attention is paid to the emergence of new absorption bands, the disappearance, decrease in intensity, or displacement of the absorption bands of the starting materials.

As expected, the intensity of the O – O bonds of the PP is significantly reduced in the product of the interaction of cellulose with PP. At the same time, changes associated with hydroxyl groups of cellulose (table 5) are observed, which gives the basis to suggest that they are involved in the initiation of these groups.

**Table 5**

Changes in the position and intensity of the absorption bands in the IR spectra during the interaction of cellulose with PP

Absorptionband, cm <sup>-1</sup>	Typeofoscillation	Atoms, bonds	Detectedchanges
1262	ν	O–O	intensityreduction
1337	δ	OH	intensityreduction
2850	ν	OH	Shifting
2928	ν	OH	Shifting

In the IR spectra of copolymers, some new bands were detected in comparison with the spectra of cellulose. An absorption band appears at 1720–1730 cm<sup>-1</sup>, referred to the C = O stretching vibrations of the AK, MMA groups and at 2241 cm<sup>-1</sup>, attributed to C≡N AN stretching vibrations. The position and intensity of the absorption bands of hydroxyl groups change. In all copolymers, there are strong absorption bands at 1027-1030, 1055-1058, medium intensity bands at 1113-1116 and 1164 cm<sup>-1</sup>. These bands relate to vibrations of simple ether bonds (Fig.). The initiation reaction of graft copolymerization of monomers to cellulose is most likely to carry out on hydroxyl groups of cellulose.

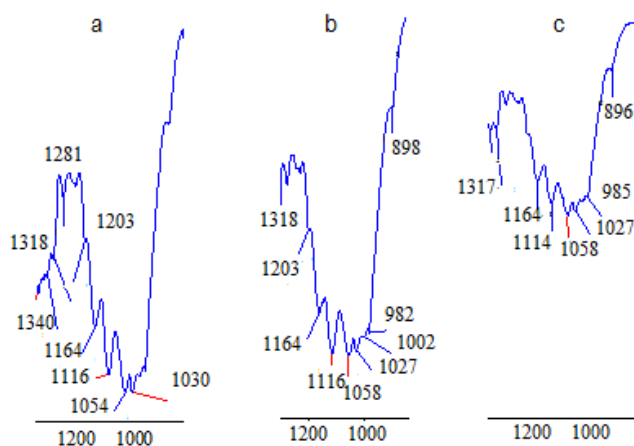


Fig. Absorption bands (ν, sm<sup>-1</sup>) vibrations of C–Obonds in the IR spectra of cellulose with AA (a), MMA (b) and AN (c)

The results of IR-spectroscopic studies allow to suggest the grafting of functionally active polymers to cellulose macromolecules. The growth of polymer chains is carried out by a radical mechanism as a result of the homolytic rupture of π-bonds of acrylic monomers.

Now we analyze the PMR spectra of cellulose and their copolymers with acrylic monomers (table 6). In the PMR spectra of cellulose, signals of chemical shifts of protons of CH, CH<sub>2</sub> groups are detected. There are multiplet signals related to protons of the OH groups in the region of 2.5-4.5 ppm. PMR signal at 6.59 and 7.68 ppm refers to the protons of CH<sub>2</sub>OH.

**Table 6**

The results of PMR - spectral studies of graft copolymers of cellulose

SignalofNMR,H, 400 MHz, NF <sub>3</sub> , δ, m. p. (intensity)	Cellulose	Copolymers	
		Cellulose- AA	Cellulose-MMA
0.37-0.48, d (2.93)	0.37-0.49, d	0.37-0.49, d (0.27)	
0.80, s (12.72)	0.80, s	0.80, s (1.07)	
1.24, q (0.88)			
1.75, s (0.79)			
2.00, s (0.42)	2.00, s	2.01, s (0.35)	
2.72, m (2.89)			
2.96, s (0.82)	3.01, s	3.03, s (1.00)	
3.24-3.33, d (0.26)	3.23-3.30, d	3.25-3.33, d (3.44)	
3.43-3.56, m (4.81)	3.56, m	3.57, m (2.19)	
3.76-3.77, d (0.48)	3.74, s	3.72, s (1.54)	
3.85, s (0.15)			
3.98, m (0.22)	3.96, s	3.92, s (0.45)	
4.18, m (1.81)	4.09-4.12-4.14-4.18, m	4.12-4.15-4.29, m (7.25)	
4.43, s (0.33)	4.31, s	4.59, s (0.15)	
	5.11, s	5.09, s (0.17)	
5.19, s (1.00)	5.21-5.26, d	5.20-5.24, d (0.22)	
6.59, s (0.12)			
7.68, s (0.10)			

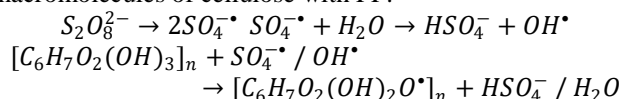
In the PMR spectra of copolymers of cellulose with AA and MMA, some changes were detected in comparison with the spectra of cellulose. Signals disappeared at 6.59 and 7.68 ppm. The number has decreased and the position of the signals has changed at 0.5-2.0 ppm. There were intense and numerous signals in the region of 2.0-5.5 ppm. The analysis showed the disappearance of some signals in copolymers (2.72 ppm), an increase (3.25 ppm) or a decrease (3.56 ppm) of the intensity of others, a shift (2.96 by 3.03 ppm, 4.19 by 4.29 ppm and 4.43 for 4.59 ppm) of thirds. Moreover, the shift is observed mainly in the region of higher values of chemical shifts. Mono signal at 5.19 ppm turns into a singlet and a doublet with maxima of 5.09, 5.20 and 5.24 ppm. New



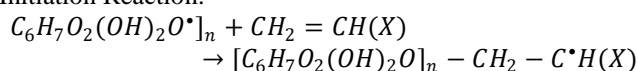
signals of PMR appear at 3.72, 4.12, 4.15, 4.29 ppm. etc.

The shift of the signals indicates to the participation of protons in the reactions of interaction between the cellulose, the initiator and the monomer. New signals are most likely to belong to the protons of graft polymers and  $OCH$  and  $OCH_2$  groups that bind it to cellulose. The observed changes in the PMR spectra of the copolymers confirm the results of the analysis of the IR spectra. The initiation of graft copolymerization is carried out through the oxygen atoms of cellulose. The growth of grafted chains occurs by a radical mechanism.

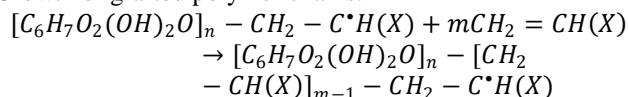
According to the analysis of literature sources and the results of IR, PMR-spectroscopic studies, the following scheme of graft copolymerization of cellulose was proposed. Formation of active centers in the interaction of macromolecules of cellulose with PP:



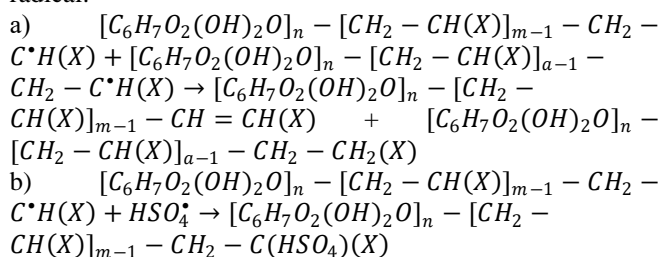
Initiation Reaction:



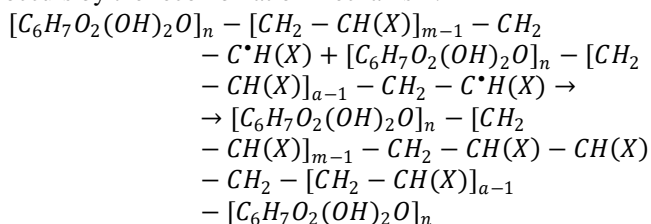
Growth of grafted polymer chains:



At low degrees of conversion, a soluble copolymer is formed. In this case, an open circuit occurs by the mechanism of disproportionation or transfer of the chain to the initiator radical:



At sufficiently deep degrees of conversion, reticulated, insoluble polymers are formed. In this case, an open circuit occurs by the recombination mechanism:



In the presented diagrams, the most probable reactions of the process of graft copolymerization of acrylic monomers to cellulose are depicted.

#### IV. FINDINGS

When potassium persulfate interacts with cellulose, active centers of a radical nature are formed, by which graft copolymerization of functionally active monomers can be carried out. Fixation of the initiator is obtained by the preliminary physical adsorption from solution and carrying out the copolymerization in the medium of a solvent that does not dissolve the initiator. The active centers of graft copolymerization appear during the heterophase interaction of the cellulose macromolecule with the potassium persulfate solution. Hydroxyl groups of cellulose are involved in the

reactions, to the oxygen atom of which a synthetic polymer chain is bound. The reaction serves in the purposeful modification of the properties of cellulose and textile materials.

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