Simultaneous Production of Cellulose Nitrates and Bacterial Cellulose

Subjects: Polymer Science

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The scientific hypothesis about the possibility of simultaneous production of two independent of each other products from Miscanthus giganteus, namely the product of chemical modification of cellulose - cellulose nitrates and the product of biosynthesis - bacterial cellulose was tested.

Keywords: *Miscanthus* × *giganteus* ; cellulose ; enzymatic hydrolysis ; cellulose nitrates ; bacterial cellulose

1. Introduction

Cellulose nitrates, being the first product of the chemical functionalization of cellulose, have been actively studied for almost 200 years [1][2][3][4][5][6][7][8][9][10][11][12][13]. The process of cellulose nitration itself, as an example of the chemical functionalization of the most widespread and easily renewable biopolymer in nature, is of particular importance for fundamental science [14]. Cellulose is a naturally created polymer of beta-glucose, the links of which are connected by a 1-4-β glycoside bond, and is produced annually in the amount of about 1.3 billion tons through photosynthesis [15]. The process of cellulose nitration consists in replacing the hydrogen atom of the hydroxyl group –H–OH of cellulose with the nitro group –NO2 under the influence of nitrating mixtures. By changing the composition of nitrating mixtures and the parameters of the nitration process, it is possible to obtain cellulose nitrates with a wide range of functional properties [1][15][16].

Due to the shortage of traditional raw materials of cotton and wood [14] in modern times, it is becoming relevant to study the possibility of obtaining new types of cellulose nitrates with regulated functional properties based on alternative sources of raw materials. The list of non-wood sources of cellulose of plant origin continues to expand actively [17][18][19][20][21][22][23][24][25][26][27][28][29].

Along with the use of cellulose of non-wood origin, the possibility of obtaining a new type of cellulose nitrates from bacterial [30][31][32][33][34]cellulose (BC) is substantiated and the prospects for its use are considered [10][35][36][37][38][39][40][41][42][43][44][45][46][47][48][49][50][51]. Additionally, there is a need to scale the biosynthesis process to ensure the precursor volumes of promising NC [52], such studies are quite rare.

An analysis of the world literature in the field of alternative sources of cellulose has shown that today, despite the variety of forms of miscanthus and the growing area [53][54][55][56][57][58][59], as well as its wide application: from the paper industry to biosynthesis products [60][61][62][63][64][65][66][67], information about miscanthus cellulose as an object of chemical functionalization into cellulose nitrates is very limited, except for with the exception of the author's individual studies [27][28][29][68].

All methods for obtaining cellulose from non-wood sources, including miscanthus, aim to meet the product requirements for the purity of cotton cellulose: the mass fraction of alpha cellulose is not less than 92%, the mass fraction of pentosans is not more than 2%, a minimum of lignin, therefore there is no information about enzymatic hydrolysis as a method of pretreatment of a cellulose-containing product to change its structural characteristics and subsequent nitration. Moreover, there are no examples of using cellulose to produce cellulose nitrates directly and, at the same time, as a source of glucose nutrient medium for subsequent biosynthesis.

2. Simultaneous Production of Cellulose Nitrates and Bacterial Cellulose

Information on enzymatic hydrolysis in order to simultaneously obtain a precursor – for the biosynthesis of cellulose nitrates and glucose nutrient medium – for subsequent biosynthesis is missing and was implemented by us for the first time.

The object of the study was nitric acid cellulose from giant miscanthus, which underwent enzymatic hydrolysis with enzyme preparations "Cellulux-A" and "Ultraflore" in an acetate buffer at an initial substrate concentration of 45.0 g/l.

At the first stage of enzymatic hydrolysis, the duration of 2, 4, 6, 8, 24, 32, 48 h changes in the degree of polymerization and the degree of crystallinity of cellulose from miscanthus gigantus, an increase in the concentration of reducing substances and a decrease in weight, in accordance with. The solid residue of cellulose from miscanthus gigantus was studied for the degree of polymerization and the degree of crystallinity of cellulose.

In the first 2 hours of hydrolysis, a decrease in the degree of polymerization of cellulose from 1770 to 1490 and an increase in the degree of crystallinity from 64% to 72% were recorded. Further, the degree of polymerization increases, reaching a maximum after 48 hours of enzymatic hydrolysis, the concentration of reducing substances reached a value of 30 g / l, which corresponds to a yield of reducing substances of 60%.

Based on the data obtained, three points of duration of enzymatic hydrolysis were selected for the second stage of the experiment: 2, 8 and 24 hours. The choice of the 2 h point is due to the maximum decrease in the degree of polymerization of cellulose, despite the fact that the concentration of reducing substances in the hydrolysate does not reach the required value for the synthesis of bacterial cellulose of 20 g/l. The points 8 and 24 h are selected, since during this time there is a change in the structural characteristics of cellulose, and the concentration of reducing substances in hydrolysates is more than 20 g/l.

The three cellulose samples obtained as a result of enzymatic hydrolysis (C2, C8, C24) differed in their characteristics from the original sample of miscanthus gigantus cellulose (degree of polymerization 1770, degree of crystallinity 64%), and also differed among themselves (degree of polymerization from 1510 to 1760, degree of crystallinity from 72 to 75%). At the same time, hydrolysates (C2, C8, C24) with different concentrations of reducing substances (from 13.4 to 27.5 g/l) were obtained.

Nitration of cellulose with a sulfuric-nitric acid mixture and analysis of cellulose nitrates were carried out similarly to clause 3.4.1. A sample of cellulose nitrates obtained from the initial cellulose of miscanthus gigantus is characterized by a nitrogen content of 12.20%, a viscosity of 120 MPa · s, solubility in an alcohol ester solvent of 41% and a high yield of 142%. An increase in the duration of enzymatic hydrolysis from 2 hours to 24 hours led to a simultaneous increase in the yield of cellulose nitrates from 116% to 131%, nitrogen content from 11.35% to 11.83% and viscosity from 94 MPa·s to 119 MPa·s. The suitability of the initial and samples of cellulose after enzymatic hydrolysis for chemical functionalization is confirmed by the extremely high 100% solubility in acetone of cellulose nitrates synthesized from them.

The SEM method shows the smoothing of roughness and preservation of the shape of the initial fibers during the nitration process with a slight increase in volume. The synthesis of cellulose nitrates was confirmed by the method of IR spectroscopy based on the presence of characteristic frequencies 1657-1659 cm-1, 1277 cm-1, 832-833 cm-1, 747 cm-1, 688-690 cm-1. TGA/DTA methods have shown that cellulose nitrates are chemically pure high-energy biopolymers (specific heat of decomposition 6.53-8.28 kJ/g).

The data obtained indicate the suitability of Miscanthus gigantus cellulose after enzymatic hydrolysis for chemical functionalization into cellulose nitrates. This approach in the production of cellulose nitrates is used for the first time in world practice.

Further, bacterial cellulose biosynthesis was performed on hydrolysates obtained after 2 hours, 8 hours and 24 hours in comparison with the control.

Determination of the number of yeast cells and acetic acid bacteria on nutrient media showed: the yeast content from 9 to 14 million CFU / ml; the content of acetic acid bacteria from 8 to 10 million CFU / ml, with the exception of hydrolysate C8 1 CFU / ml, due to the low concentration of reducing substances 13.4 g / l. The yield of bacterial cellulose after 8 hours and 24 hours was at a high level – 11.1% and 9.6%, respectively. Bacterial cellulose samples had a mesh structure of intertwined fibrils, while the average width of microfibrils obtained on C8 hydrolysate was 65.0 nm, on C24 – 81.0 nm, the degree of polymerization of the obtained bacterial cellulose samples was 2100 and 2300, respectively. Thus, it was found that nutrient media based on hydrolysates obtained after 8 h and 24 h are benign for obtaining high-quality bacterial cellulose samples.

This approach of simultaneous production of cellulose nitrates and bacterial cellulose is used for the first time, tested on the lignocellulose of the energy plant – Miscanthus gigantus, the results obtained have no world analogues.

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