*Chemistry of Natural Compounds, Vol. 34, No. 3, 1998*

# DEGREE OF POLYMERIZATION AND POLYDISPERSITY OF CELLULOSE OBTAINED FROM CYCLONE FLUFF BY THE OXYGEN-SODA METHOD

**R. Saifutdinov**

UDC 661.728.21.08

*The dependence of the degree of polymerization and the polydispersity of samples of cellulose isolated from cyclone fluff under the conditions of an oxygen-soda cook has been studied. It has been shown that the cellulose obtained from soda-oxygen digestion in the presence of copper sulfate and hexamethylenetetramine is more monodisperse and has a higher degree of polymerization. Films and fibers of the triacetylcellulose obtained from this cellulose possess the highest physicomechanical properties.*

We have previously shown the possibility of obtaining high-quality cellulose from various types of lint and cyclone fluff [1, 2].

In the present paper we give the results of a determination of the degrees of polymerization (DPs) and molecular mass distributions (MMDs) of samples of cellulose obtained by the soda-oxygen digestion of cyclone fluff with the addition of degradation inhibitors.

Soda digestion is the mildest method of isolating cellulose; however, the presence of molecular oxygen promotes the destruction of its macromolecules and the cellulose obtained has a comparatively low DP. If the soda-oxygen digestion of cyclone fluff is performed with the addition of degradation inhibitors – hexamethylenetetramine or triethanolamine – the DP of the cellulose obtained is higher (Table 1).

The polydispersity of the macromolecules of the cellulose samples was investigated by velocity sedimentation in an ultracentrifuge. The molecular composition was characterized by distribution functions (in differential, $q\% = f(S)$, and integral, $W\% = f(S)$, forms (Fig. 1)). A comparison of curves *1* and *2* in Fig. 1 shows that the addition of copper sulfate and triethanolamine to a soda-oxygen cook led to a shift of the distribution curve in the direction of higher molecular masses. At the same time, the geometric width of the distribution was somewhat greater. The MMD curve for cellulose obtained by a soda-oxygen cook without additives was located in the region of lower molecular masses. In the case of the soda-oxygen cook with the addition of copper sulfate and hexamethylenetetramine there was a considerable decrease in the amounts of low- and high-molecular mass fractions of the cellulose and a considerable rise in the uniformity of its fibers (curve 3).

In order to determine the suitability for chemical processing of the cotton cellulose obtained by the technology developed, experiments were conducted on the production of triacetylcelluloses. The physicochemical characteristics of the TAC syrups obtained are given in Table 1.

As can be seen from Table 1, the highest quality indices were possessed by a TAC syrup obtained from cellulose isolated by a soda-oxygen cook of cyclone fluff with the addition of copper sulfate and hexamethylenetetramine. A relatively low quality index was observed for the TAC syrup from cellulose obtained by a soda-oxygen cook without additives. It may consequently be assumed that the quality index of a TAC syrup depends on the DP and polydispersity of the initial cellulose.

To evaluate the dependence of the physicomechanical properties of articles from TAC on the DP and the MMD of the initial cellulose, films were cast and fibers were formed from these syrups. The results of measurements of their physicochemical properties are given in Table 2. As can be seen from Table 2, the greatest elasticity and mechanical strengthwere possessed by the films and fibers from the TAC derived from the cellulose obtained by a soda-oxygen cook with the addition of copper sulfate and hexamethylenetetramine. This is apparently explained by the fact that the initial cellulose had the highest DP and was acetylated uniformly.

---

Tashkent Institute of Chemical Technology. Translated from Khimiya Prirodnykh Soedinenii, No. 3, pp. 368-370, May-June, 1998. Original article submitted March 2, 1998.

0009-3130/98/3403-0331$20.00 

TABLE 1. Conditions for Obtaining Cellulose and Physicomechanical Characteristics of the TAC Syrups Produced from It

| Conditions of the soda-oxygen cook of cyclone fluff | Degree of polymerization of the cellulose | Characteristics of the TAC syrup | | |
|---|---|---|---|---|
| | | transparency, mm | filterability, $cm^3$/70 min | viscosity, Pas |
| 1. Without additives | 1633 | 51.9 | 291 | 29.5 |
| 2. With the addition of copper and triethanolamine | 1920 | 68.1 | 285 | 34.2 |
| 3. With the addition of copper and hexamethylenetetramine | 1547 | 74.5 | 342 | 37.1 |

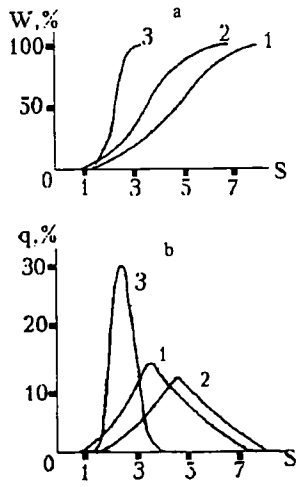


Fig. 1. Integral (*a*) and differential (*b*) MMD curves of samples of cellulose obtained under various conditions of a soda-oxygen cook: *1*) without additives; *2*) with the addition of copper sulfate and triethanolamine; *3*) with the addition of copper sulfate and hexamethylenetetramine.

Thus, the cellulose obtained by a soda-oxygen cook with the addition of copper sulfate and hexamethylenetetramine had a high uniformity of the macromolecules, and films and fibers from the TAC it produced possessed higher physicomechanical properties.

## EXPERIMENTAL

The DPs of the samples of cellulose were determined by measuring the relative viscosities of their cadoxene solutions with the aid of an Ubbelohde viscometer [3].

The MMDs of the cellulose macromolecules were determined by precipitation in an ultracentrifuge by the velocity sedimentation method [4-6].

**Preparation of the TACs.** One part of cellulose with a moisture content of 7-8% was treated with 2.4 parts of glacial acetic acid, and the mixture was stirred at 38°C for an hour. Then a solution consisting of four parts of glacial acetic acid [sic] was added. Stirring was continued at the same temperature for 45 min, after which the mixture was cooled to 18°C, and 2.7 parts of 98% acetic anhydride and 6.12% (on the weight of the cellulose) of concentrated sulfuric acid were added. With stirring, the temperature was gradually raised to 32-35°C over 2-2.5 h. A mixture of one part of water and two parts of acetic

TABLE 2. Physicomechanical Properties of TAC Films and Fibers

| Quality index of the TAC syrup | Film | | | | | Fiber | |
|---|---|---|---|---|---|---|---|
| | [η] | breaking strength, kgf/cm$^2$ | breaking elongation, % | number of double bends | breaking strength, km | breaking elongation, % | number of double bends |
| 8.5 | 1.6 | 680 | 5.7 | 115 | 11.9 | 23.9 | 2790 |
| 9.7 | 1.96 | 730 | 7.9 | 161 | 12.1 | 25.4 | 3020 |
| 12.7 | 2.20 | 750 | 8.3 | 184 | 14.7 | 26.3 | 3270 |

acid was added to the resulting viscous and transparent syrup over an hour. The TAC syrup so formed was filtered through two layers of calico in a special apparatus.

## REFERENCES


1. T. M. Mirkamilov, R. S. Saifutdinov, M. A. Askarov, and A. Abidkhanov, USSR Inventors' Certificate No. 587,192; Byull. Isobret., No. 1 (1978).
2. T. M. Mirkamilov, G. L. Akim, and R. S. Saifutdinov, USSR Inventors' Certificate No. 910,899; Byull. Izobret., No. 9 (1982).
3. Practical Handbook on the Chemistry and Physics of Polymers [in Russian], Khimiya, Moscow (1977).
4. V. N. Tsvetkov, V. E. Éskin, and S. Ya. Frenkel', The Structure of Macromolecules in Solutions [in Russian], Nauka, Moscow (1964), p. 719.
5. S. Ya. Frenkel', Introduction to the Statistical Theory of Polymerization [in Russian], Nixie, Moscow–Leningrad (1965), p. 226.
6. S. R. Rafikov, S. A. Pavlova, and I. I. Tverdokhlebova, Methods of Determining Molecular Weights and Polydispersities of High-Molecular-Mass Compounds [in Russian], Nauka, Moscow (1963), p. 335.