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Regenerated Cellulose Fibres After Controlled Enzymatic Hydrolysis

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Abstract

Controlled enzymatic hydrolysis of cuprammonium and various types of viscoses was undertaken to gain some insight into the heterogenity of the structure from the surface to core. Electron microscopic study of the hydrolysed samples at various stages of hydrolysis, also gives general idea of the morphological differences in these regenerated celluloses. The difference in the physical and especially the mechanical properties of the different types of these fibres can now be more definitely correlated with their structure.

Experimental

Four types of regenerated cellulose fibres used in this work are given in Table 1. Hydrolysis was carried out by cellulase obtained from Trichoderma Viride (activity C_1 - 183400 gm of glucose released/gm of solid enzyme and C_x - 9200 gm of glucose released/gm of solid enzyme). The strength of enzyme solution was kept 1% on the weight of fibre for all investigations. The samples were incubated in 25 ml stoppered volumetric flask with required amount of enzyme in 0.5 M acetate buffer (pH 4.8) at 48°C. After various intervals, the samples were taken out, solution diluted and the glucose released estimated colorimetrically by adding previously calibrated 3, 5 di-nitro salicylic acid solution (1). The undigested sample was washed and examined by Scanning Electron Microscope after usual sample preparation.

Results and Discussion

Figure 1 shows the amount of glucose released during the hydrolysis of different samples under consideration. The rate of hydrolysis is principally governed by the rate at which enzyme diffuses and reacts from the surface to the core and hence is a measure of compactness of the different constituent layers. In cuprammonium, the initial and the final rate is high and inbetween there is a region of slower rate (between 7-17 hrs) of hydrolysis. In viscose, however,the initial rate is much slower followed by a faster rate but there is no marked slow down as observed in the case of cuprammonium. The hydrolysis is complete in about 30 hrs in cuprammonium and

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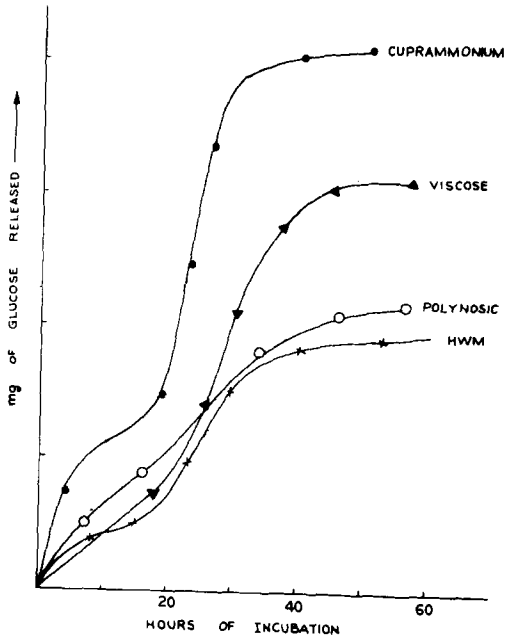

Figure 1: Enzymatic hydrolysis of celluloses

TABLE 1

	Type	Trade name	Manufacturer
1)	Viscose	Gwalior viscose	Gwalior Rayons, India
		Bodanella	Feldmühle Ltd., Switzerland
		Brenka	British Enka, Ltd., U.K.
2)	Polynosic	Zantrel	American Enka, U.S.A.
		Tufcel	Toyobo Japan
3)	High wet modulus	Grasilene	Gwalior Rayons, India
4)	Cuprammonium	Cupresa	Farbenfabriken, Bayer A.G. Germany

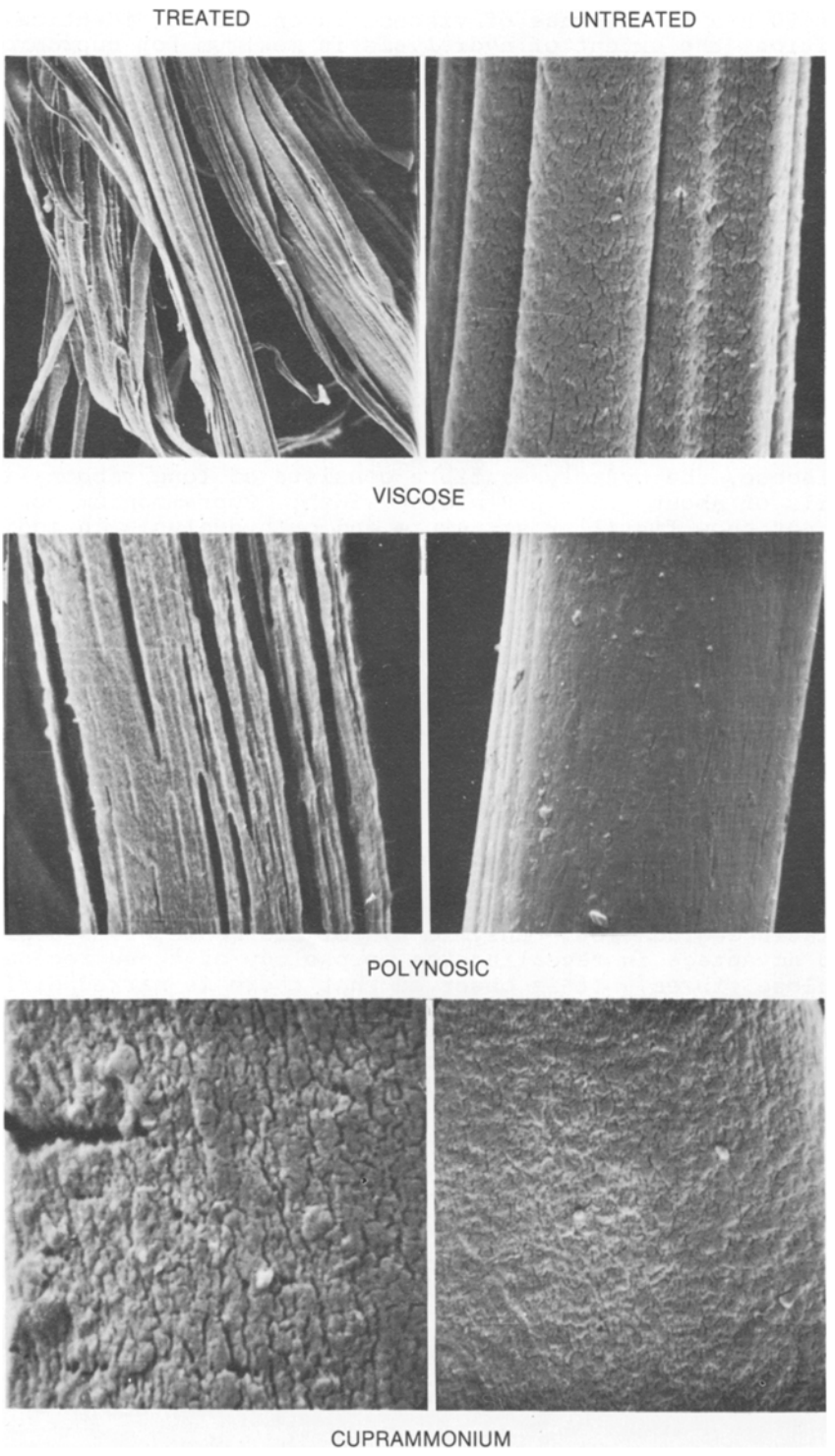

Figure 2: Scanning Electron Micrographs (magnification 7000 X)

about 50 hrs in the case of viscose rayon. Under identical conditions the extent of hydrolysis is maximum for cuprammonium and least for polynosic and HWM.

It is hence quite obvious that the cuprammonium has a soft outer layer and core compared to viscose but has a compact layer between the two soft layers which acts as a barrier. While in the viscose rayon the skin is more compact and the structure is less compact in core. On the contrary most of polynosic and HWM show all-skin structure.

Microscopic examination of the samples after successive stages of enzymatic hydrolysis is expected to reveal more information about the morphology of the samples under consideration. In Fig 2 are reproduced Scanning Electron Micrographs of the untreated and samples left when the hydrolysis is practically complete. It is interesting that the nature of degradation is different in all the three samples. In viscose, the hydrolysed fibre consists of long ribbon like fibrils of about 0.5 - 0.8 micron width. Cuprammonium however, does not show fibrillar structure and on hydrolysis in addition to longitudinal, lateral cracks develop showing the path through which the enzyme diffuses inside and hence disposition of the weak places in the fibre. Difference in the mechanical strength of the two fibres is hence very clearly seen to arise from the observed morphological differences.

Since the cellulose fibres are insoluble molecules with great structural complexity, the physical association with the enzyme can only be achieved by diffusion of these enzyme molecules to the susceptible sites on the gross surfaces of the fibre or the molecular surfaces within the fibre wall. Thus, the structural features of the fibre or its constituents that limit its accessibility or the diffusion of cellulolytic enzymes in the close proximity to that fibres will exert a profound influence on the susceptibility or resistance of the fibre to enzymatic degradation. Enzymic hydrolysis hence gives a pronounced advantage in revealing the morphology of these regenerated cellulose fibres. It is observed that there is marked difference in the structure of viscoses and cuprammonium which reflects the different mode of cellulolytic attack. Though the origin of the ribbon-like fibrils in the viscose rayon is not yet very clear, the morphology obviously depends on the mode of digestion of the cellulose so also the physical and chemical conditions of coagulation and spinning. Further work to elucidate these aspects is in progress.

Reference

1) SUMNER, J., SOMMERS, C., Laboratory Experiments in Biological Chemistry, 1st Edn., New York, Academic Press Inc., 1944

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