BRIEF COMMUNICATIONS

USE OF THIN-LAYER CHROMATOGRAPHY FOR DETERMINING SUGARS IN MICROCRYSTALLINE CELLULOSE

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Recently, microcrystalline cellulose (MCC) has found use in pharmaceutics and medicine, and, therefore, the estimation of the degree of its chemical purity is acquiring first-degree importance. Usually, the destruction of natural cellulose takes place on heterogeneous hydrolysis in the presence of an acid in an aqueous medium followed by the washing of the final product (MCC) with water to a neutral reaction [1, 2].

We have used the TLC method for determining sugars sorbed by microcrystalline cellulose in these media.

A 0.2-g weighed sample of cotton MCC obtained under laboratory conditions by the degradation of cotton cellulose in aqueous organic media was washed with 30-40 ml of distilled water (steeping) on a Schott No. 1 filter. The filtrate was dried in vacuum, and the weight of dry residue was determined. This was dispersed in 1 ml of water, and the concentration of the solution so prepared was between 0.02 and 0.09 g/ml.

TLC was performed on prepared 5 × 10-cm plates with layers of KSKG silica gel (pore diameter ~120 Å; particle diameter 5-17 μm) with silica sol as binder [4]. From 1 to 5 μl of solution was deposited on each plate. Chromatography was carried out by the ascending method (h = 8 cm). The eluent used was ethyl acetate–isopropanol–water (5.0:3.2:1.9). The plates were dried at 100°C for 10 min. Oleum vapor was used for detection, the plates being exposed to it for 1-2 min and then being heated at 180°C for 30-40 min until distinct dark spots had appeared.

The following sugars were used as standards: lactose (R_f 0.23), raffinose (R_f 0.10), cellobiose (R_f 0.30), D-xylose (R_f 0.71), D-glucose (R_f 0.52), galactose (R_f 0.43), sucrose (R_f 0.34), D-fructose (R_f 0.49), rhamnose (R_f 0.48), arabinose (R_f 0.54), and levoglucosan (R_f 0.79).

In all cases, in extracts from MCC we identified from their R_f values raffinose, cellobiose, D-glucose, D-xylose, and levoglucosan. We may note that unidentified spots were present at the start which probably consisted of oligomeric products of the degradation of the cellulose. Above the starting line appeared two horseshow-shaped spots with R_f 0.05 and 0.09 which we identified as galacturonic and glucuronic acids.

The amount of sugars (c) was calculated from the relationship (2a)(2b) → f(c) obtained for the reference sample, where a and b are the dimensions of the spot in the vertical and horizontal directions. As an example, Fig. 1 shows the relationship (2a)(2b) = f(c) and

TABLE 1

Weight of MCC for extraction, g	Loss in wt. after extraction, % on the initial weight	Sugars identified, % on the initial weight					
		raffinose	cellobiose	D-glucose	D-xylose	levoglucosan	total
0,2028	9,07	1,6	3,1	1,3	0,8	1,0	7,9
0,1958	10,61	2,5	2,2	2,7	0,8	1,2	9,4
0,2153	8,91	1,4	2,0	1,2	0,8	2,7	8,1
0,1717	12,23	1,3	3,2	1,4	1,0	3,9	10,8

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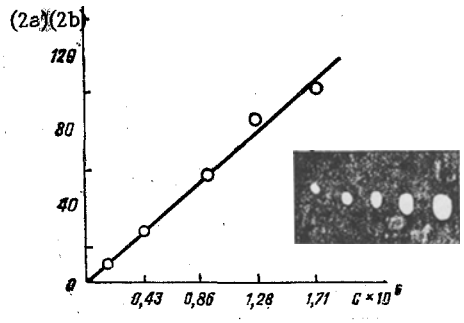

Fig. 1. Dependence of the dimensions of the spots on a chromatogram on the amount of cellobiose, and a chromatogram of different amounts of cellobiose; a, b) dimensions of the spots in the vertical and horizontal sections, mm; c) amount of substance, g × 10^{-6}.

a chromatogram of different amounts of cellobiose.*

Table 1 shows the results obtained for the amounts of sugars by the TLC method for the case of a number of samples of MCC degraded under various conditions. The total amount of sugars determined in the extracts was somewhat less than the loss in weight after extraction. However, in this total no account was taken for the small amount of uronic acids, identified quantitatively but not subjected to quantitative estimation, and of the oligomeric products remaining at the start. The sensitivity of the method amounts to 0.4-2.0 μg of substance per spot and, consequently, it may be considered satisfactory as a semiquantitative estimate.

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*In [3] the linear relationship log c = f(2a)(2b) is shown, which is possible for a small range of concentrations.