

EFFECT OF AQUEOUS PERCHLORIC ACID ON THE XYLAN CONTENT OF HOLOCELLULOSE FROM CORNSTALKS^{1,2}

EMMETT BENNETT

MASSACHUSETTS AGRICULTURAL EXPERIMENT STATION,
AMHERST, MASSACHUSETTS

Hemicelluloses are not easily removed completely from plant material by any solvent. Preliminary tests in this laboratory indicated that isolated hemicelluloses were reasonably soluble in aqueous perchloric acid. Because of this observation, the difficulties in extraction, and the fact that this solvent is satisfactory for starch (1, 2) it was of interest to determine the possibilities of perchloric as a solvent for hemicelluloses.

Holocellulose prepared from cornstalks (3) was used as source material. Five molar and 2.5 M perchloric acid were used for extractions. The content of furfural calculated as xylan and the loss in total weight of the samples were used to indicate the progress of the reaction which was carried out as follows: a 0.35 gm charge of holocellulose in triplicate was weighed in tubes 28×160 mm and placed in a constant temperature water bath operating at 25° C. The tubes were equipped with a motor-driven stirrer which operated during the entire solubility test. Fifteen ml of 5 or 2.5 M perchloric were added from a pipette while the sample was stirred. Solubility tests were conducted for 15, 30, 45, 60, 90 and 120 minutes. Control samples were obtained by extracting the sample with water for the same period of time. At the end of each trial the sample was filtered and washed with ethanol, ether and dried. The residues were weighed, composited and subjected to the furfural test according to the AOAC procedure (4).

The data are shown in graphic form in figure 1. Solution and precipitation of hemicelluloses are usually associated with basic and acidic media, respectively. These data, however, indicate that 5.0 M perchloric acid is positive but not selective in action, that the removal of total solids proceeds at a faster rate than the removal of xylan, that the maximum loss of total solids is only about 80 % of the xylan present, that the xylan removed constitutes approximately 68 % of the total loss, and finally that after two hours of contact less than 60 % of the xylan has been removed. A 2.5 M solution was less effective as a solvent but followed a similar pattern. Complete extraction of xylan by 5.0 M acid would require several hours at best. In the meantime it is almost certain that considerable hydrolysis would have occurred. It is believed, however, that holocellulose affords a highly suitable substrate for the solubility tests inasmuch as the preliminary treatments removed some encrusting substances. Nevertheless, the data indicate that the celluloses are firmly bound and that this removal is accompanied by loss of hexosan. The data presented here and elsewhere (5) indicate that a satisfactory

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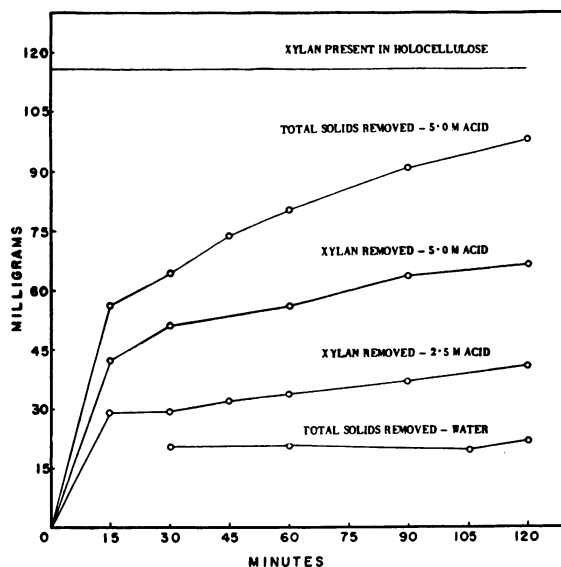


FIG. 1. The weight of total solids and xylan removed from cornstalk holocellulose by aqueous perchloric acid at 25° C for periods up to two hours.

removal of furfural-yielding substances from most celluloses is perhaps unattainable and really undesirable by chemical means. The problem is really to fractionate on the basis of chain lengths rather than on a qualitative basis.

SUMMARY

Hemicelluloses are soluble in aqueous perchloric acid. The total solids removed by 5.0 M perchloric acid were about two-thirds xylan and one-third hexosan; about 60 % of the xylan was removed, thus indicating the positive but nonselective and incomplete solvent action of 5.0 M perchloric acid.

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