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Author(s)	SHIMIZU, Kazumasa; ABE, Fusako; ISHIHARA, Mitsuro; MIYAZAKI, Kango
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Effect of the Thermal Treatment on Wood Hemicelluloses VIII†

Thermal Analysis of Cellulose Containing Xylan*

By

Kazumasa SHIMIZU**, Fusako ABE**, Mitsuro ISHIHARA**
and Kango MIYAZAKI**

木材へミセルロースに及ぼす熱処理の影響 (第8報)†

キシランを含有するセルロースの熱分析*

志水一允** 阿部房子**

石原光朗** 宮崎鑑吾**

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Introduction

It is well known that hemicellulose dissolved in cooking liquor are redeposited on the fibres during kraft pulping^{1,2)}. The sorbed hemicellulose is important since it significantly affects the properties of the final paper products³⁾. The sorption of hardwood 4-*O*-methylglucuronoxylans has been extensively studied under the conditions of alkaline pulping⁴⁻⁹⁾.

On the other hand, among the main components of wood, hemicellulose is attacked first and cellulose is considerably more stable on heating. Thermally, the most resistant component is lignin. And the wood thermogram has been shown to be a summation of the thermograms of the individual wood components, although some deviations were observed¹⁰⁻¹⁵⁾. This kind of deviation has been

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** 農林省林業試験場 Government Forest Experiment Station, Meguro, Tokyo.

explained by the interactions among the wood components. SAWABE et al.¹⁶⁾ suggested that the thermal degradative reaction of cellulose is affected by the presence of hemicellulose and that the effect for Hinoki is negative-catalytic, but positive for Buna.

The purpose of this study is to investigate the effects of sorbed xylan on the thermal behavior of cotton cellulose by means of TG and DTA.

Experimental

1. Samples

a. Hardwood xylan

Shirakaba (*Betula platyphylla*) was milled to a 100–120 mesh meal, exhaustively extracted with methanol and then water for 2 days with occasional stirring. A crude xylan was isolated by direct extraction of the extractive-free wood meal with 10% aqueous potassium hydroxide with a yield of 15.5% in the same way as described in a previous paper¹⁷⁾. The crude xylan (30 g) was purified by dissolution in 5% aqueous potassium hydroxide (2 ℓ) and precipitation by the addition of ethanol (1.5 ℓ)¹⁷⁾. The precipitated xylan was recovered by centrifugation, washed successively with 80% aqueous ethanol, absolute ethanol and ethyl ether and dried in vacuum over phosphorus pentoxide (yield, 20.5 g).

Hydrolysis and examination by gas liquid chromatography (GLC) as alditol acetate revealed only a small amount of rhamnose except xylose. Analysis: Methoxyl: 2.56%, Uronic anhydride: 9.7%, Klason lignin: 0.63%, Acid soluble lignin: 0.92%, Intrinsic viscosity in cadoxen: $73.0 \text{ cm}^3/\text{g}^{18)}$, corresponding to DP_w 183.

Before use the xylan (5 g) was treated with 1.0 g of sodium borohydride in 250 ml of water for 3 hours at room temperature. The liquor was neutralized with acetic acid and poured into two volumes of ethanol. The reduced xylan was recovered in the same way as described above.

b. Cotton cellulose

The cotton cellulose used was for medical use and was cut to a fibre length of about 3 mm. On hydrolysis, it afforded glucose and a trace amount of xylose.

Reduction of the cotton cellulose was carried out in 0.2 M sodium borohydride solution for 48 hours at room temperature at a cellulose-liquid ratio of 1:100¹⁹⁾.

2. Performance of sorption experiment

The sorption experiments were carried out following the method of HANSSON et al.^{8,9)} The xylan (600 mg) was dissolved in a 1.5% sodium hydroxide solution (12 ml) in glass ampoules which were filled with nitrogen. Then, the cotton cellulose (300 mg) was added.

The ampoules were sealed and placed, surrounded by water, in small stainless steel autoclaves which were immersed in a temperature regulated oil bath (170°C). The time was recorded from 2 min after immersion in the bath.

After sorption the cotton cellulose was filtered and washed thoroughly with 0.2% sodium hydroxide solution, hot water, 1% acetic acid solution, and finally

with cold water. The washed cotton cellulose was dried in air for 2 days and in vacuum over phosphorous pentoxide.

The filtrate was acidified to pH 4 with acetic acid and poured into four volumes of ethanol. The precipitate formed was centrifuged, washed and dried in the usual way.

3. Preparation of the blended samples

The physical blends of xylan and cotton cellulose were prepared by suspending the xylan in water, mixing in the cellulose fibres and freeze-drying. The amount of blended xylan was calculated by the increased weight of cotton cellulose.

4. DTA and TG

The thermal analysis was conducted in an apparatus supplied by Shinkuriko Company, by which the weight loss and the temperature change of the reactant in a sample cell can be measured simultaneously. The sample (40 mg) was packed into a platinum crucible and heated from room temperature to 400°C at a rate of 2.5°C/min in air, nitrogen stream (at a rate of 40 ml/min) and vacuum (below 1.0 mm Hg).

5. Determination of carbohydrate composition

The carbohydrate composition was determined by hydrolysis to constituent monosaccharides, preparation of the alditol acetate derivatives and determination of the amount of these derivatives by GLC^{20,21}. Inositol was used as an internal standard.

The relative amounts of the monosaccharides in a hydrolyzate were obtained by measuring the areas of the peaks of the compounds in the chromatogram by a Disk chart integrater and reading the corresponding amounts of the sugars from the calibration curve.

The chromatograms were recorded with a Shimazu GC-1C gas chromatograph equipped with a double column and a hydrogen flame detector. The stainless steel column was 185 cm long and 3 mm in diameter and was filled with Gascrom Q, 100-120 mesh, containing 3% ECNSS-M. The conditions were: injection port temperature 250°C, column temperature 180°C, detector temperature 250°C. The flow rate of the carrier gas, nitrogen, was 60 ml/min.

6. Viscosity measurement

The weight average degree of polymerization (DP_w) of the xylan was calculated from the intrinsic viscosity measured in cadoxen solution according to the procedure used by WIKSTRÖM¹⁸.

The number average degree of polymerization (DP_n) of the cotton cellulose was calculated from the intrinsic viscosity data by the method of JAYME et al.²² and SMITH et al.²³

7. Determination of uronic anhydride

Uronic anhydride content was estimated by the decarboxylation method following the procedure of JOHANSSON et al.^{24,25}

Results and discussion

The sorption experiment of xylan on cotton cellulose was carried out by the method of HANSSON et al.^{8,9)} using 1.5% sodium hydroxide solution at 170°C. The cotton cellulose samples containing the sorbed xylan were prepared by the treatment for 10, 25 and 40 min. They were designated as CX-10, CX-25 and CX-40, respectively. The amount of the sorbed xylan increased with an increased time (Table 1).

The unadsorbed xylan was recovered from the sodium hydroxide solution used in the sorption experiment by the addition of ethanol after neutralization with acetic acid. The analytical data for the recovered xylan are summarized in Table 1. It is well known that polysaccharides are chemically degraded by the peeling reaction and the alkaline hydrolysis during the treatment with alkali. This is particularly true of hemicellulose^{26,27)}. Some polysaccharides are stabilized to the endwise type of alkaline degradation by the reduction of the reducing terminal sugar unit into a non-reducing sugar alcohol unit with borohydride. In this experiment the cotton cellulose and xylan used were reduced with borohydride before the sorption experiment in order to suppress the degradation as much as possible. As can be seen from Table 1, the recoverable amount of xylan decreased and its DP_w was lowered with prolonged time in spite of the pretreatment with borohydride. And as expected, 4-*O*-methylglucuronic acid was partially removed during the treatment with alkali. These results are in good agreement with earlier investigations^{26,27)}. For comparison, cotton celluloses were treated with alkali under the same condition without the presence of xylan. They were designated as C-10, C-25 and C-40 according to the treatment times. The changes in DP_n were slight, as shown in Table 1. The dynamic TG and DTA curves for these treated celluloses in vacuum shifted in parallel to higher temperature sides (at most 3°C) compared to those of the untreated one (C-0) (Fig. 1). The thermograms of other samples, C-10 and C-25 lay between C-0 and C-40. This trend was the same in the other atmospheres, air and the nitrogen stream. This may be due to the removal of impurities or changes in the physical state of cellulose with alkaline treatment. C-40 was regarded as a control sample.

The DTA and TG curves for C-40, CX-10 and CX-40 in different atmospheres

Table 1. Result of Sorption Experiments

Treating time (min)	Cellulose* DP_n	Sorbed xylan (%)	Recovered xylan			
			Amount (%)	DP_w	Uronic anhydride (%)	Methoxyl (%)
0	1420	—	—	183	9.7	2.56
10	1373	1.3	80.7	182	8.6	2.34
25	1369	3.0	74.6	167	7.7	1.94
40	1329	4.5	55.3	128	6.8	1.52

* The cellulose was treated without the presence of xylan

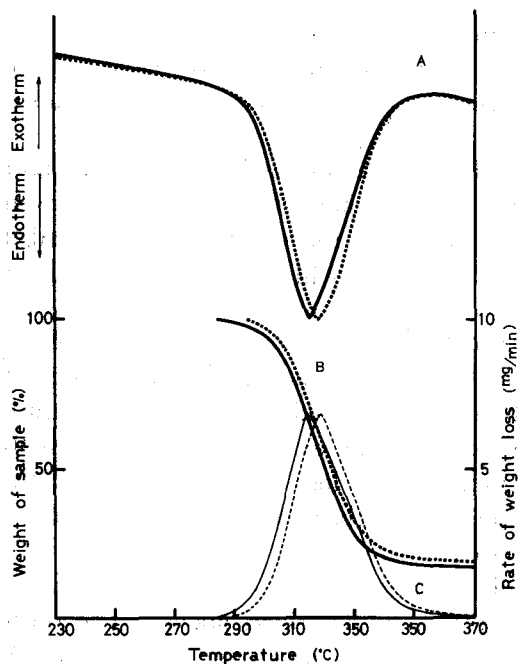


Fig. 1. Thermograms of cellulose treated with alkali in vacuum
 —: C-0, ----: C-40
 A: DTA, B: TG, C: differential curve derived from TG

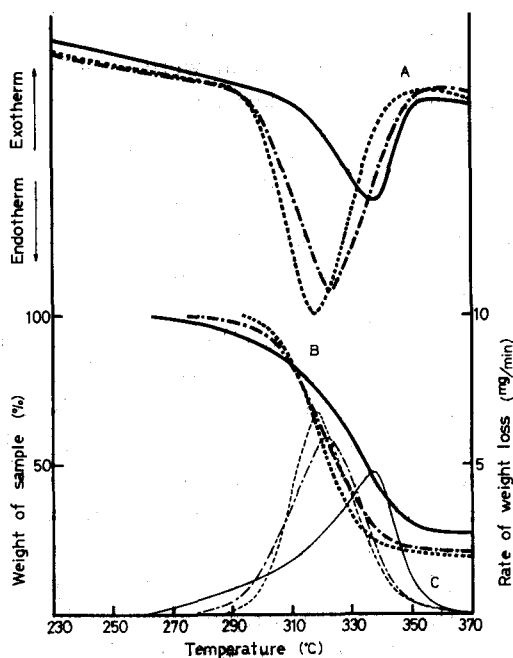


Fig. 2. Thermograms of cellulose containing sorbed xylan in vacuum
 ----: C-40, - · - · -: CX-10, —: CX-40
 A: DTA, B: TG, C: differential curve derived from TG

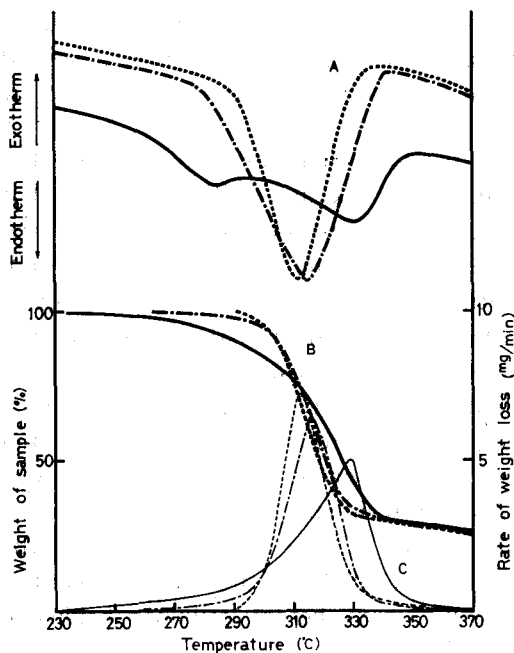


Fig. 3. Thermograms of cellulose containing sorbed xylan in nitrogen stream
 legend: see Fig. 2.

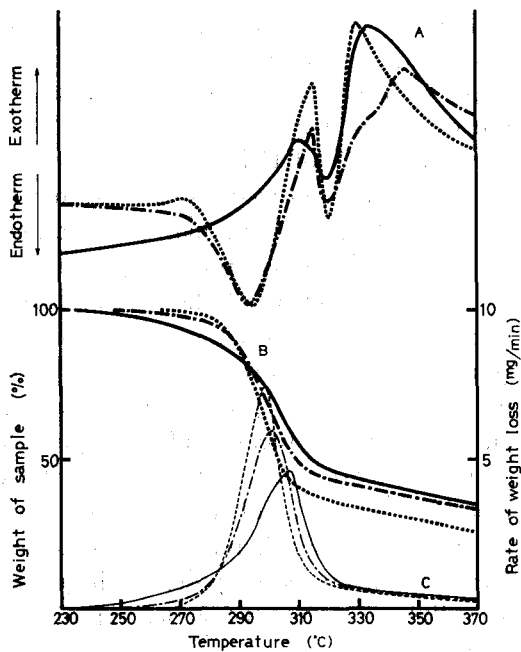


Fig. 4. Thermograms of cellulose containing sorbed xylan in air
 legend: see Fig. 2.

are shown in Figs. 2 to 4. The thermograms for CX-25 were between those for CX-10 and CX-40, and were excluded from the figures to avoid complexity. The differential curves derived from the TG curves in each atmosphere are also shown in these figures.

In vacuum (Fig. 2), weight loss of C-40 began at 294°C and the rate of weight loss rapidly increased, reaching a maximum at 318°C where the DTA curve showed the nadir, continuing until about 333°C. Beyond 333°C, the weight loss curve tailed off and showed slow decomposition of residual char of which the yield was about 19%.

But CX-10 and CX-40 in vacuum started to decompose at lower temperatures and at slower rates compared with those of C-40. The maximum rate of weight loss and the endothermic nadir of both the samples moved to higher temperature with an increase in the quantity of sorbed xylan. For example, those of CX-40 were 20°C higher than of C-40. And the rapid weight loss was complete at 350°C leaving 28% char. With an increased quantity of sorbed xylan, the DTA curves showed wider endotherms and more decreased areas.

The DTA and TG of the cotton cellulose containing the sorbed xylan in air and the nitrogen stream showed the same trends as described above. The thermograms in nitrogen stream were similar to those in vacuum (Fig. 3.) The DTA curve of CX-40 showed two endotherms with the nadirs at 285°C and 330°C indicating that the former was attributable to the decomposition of the sorbed xylan.

The DTA curves of C-40 in air showed a first deep endotherm with the nadir at 296°C corresponding to the rapid weight loss of TG curve (Fig. 4). This endotherm is masked by a following sharp exotherm having the peak at 315°C. Up to this temperature, the major weight loss nearly terminated. Then, a second sharp endotherm appeared, which had the nadir at 322°C, and was followed by the exotherm with the peak at 330°C.

The DTA of CX-40 in air showed that the first endotherm observed in that of C-40 was replaced by an exotherm with the peak at 312°C indicating that the initial stage of thermal degradation of cellulose was severely affected by the presence of xylan as observed in the thermograms in vacuum and nitrogen stream.

The mechanism of the thermal degradation of cellulose has been studied quite frequently during recent years. There is general agreement that the degradation consists of the following competing reactions²⁸⁾: (1) a dehydration of the cellulose by an endothermic process to produce dehydrocellulose, (2) a depolymerization by an endothermic process and subsequent levoglucosan formation, (3) a decomposition of the dehydrocellulose formed in the first process via one or more exothermic reactions into a number of gaseous products and residual char.

On the other hand, as reported in the earlier studies of this series²⁹⁻³¹⁾, the hardwood xylan decomposes in the range of 200° to 290°C by the same mechanism as cellulose. The major decomposition of xylan ends before cellulose begins to decompose.

It is obvious from the results described above that the initial stage of degradation of the cotton cellulose was affected by the presence of xylan, and that the

cellulose decomposed at a slower rate. Therefore, it can possibly be considered from these facts that since the rapid and large loss of weight from cellulose is caused by the second process, this process is restrained by an interaction between the cellulose and xylan.

The mechanism of the second process is explained by the following steps^{31,32}: (1) an initial chain scission of cellulose molecules whereby thermally unstable chain ends are formed (initiation reaction), (2) an unzipping of monomer units from these unstable chain ends (propagation reaction), (3) a stabilization of chain ends, thus terminating the splitting off of monomer units (termination reaction).

Although sufficient evidence has not been obtained, the interaction is presumed to promote the termination reaction shortening the kinetic zip length and suppressing the rapid weight loss.

Furthermore, the sorbed xylans are more resistant to cold alkali extraction and to acid hydrolysis^{1,6} than the xylan of holocellulose or sulfite pulp, indicating that it is partly fixed to the cellulose through glycosidic linkages³³, co-crystallization³⁴ and/or hydrogen bonds^{5,8}. The influence of xylan on the thermal degradation of cellulose is expected to be different depending on the state of xylan in cellulose.

Therefore, the physical blends of xylan and cellulose were prepared by suspending the xylan in water, mixing in the cellulose fibres and freeze-drying. Thus, both the samples were blended as homogeneously as possible and the formation of a hydrogen bond between the samples was avoided.

The thermal analyses of these blends samples were conducted in the same way as the sorption system. The results are shown in Figs. 5 to 7.

The TG curves in all atmospheres exhibited slightly different patterns from those of the sorbed samples. That is to say, the TG curves of the blends showed two distinct ranges attributable to the decomposition of each component. The cellulose with the sorbed xylan showed a smoother S-shaped weight loss curve than the blends, suggesting that the former had more intimate contact between cellulose and xylan than the latter.

Nevertheless, the DTA curves of the blends in vacuum and the nitrogen stream showed that the endothermic nadir corresponding to the maximum rate of weight loss moved to the high temperature side. In air, the first endotherm for C-0 was replaced by an exotherm in the blends. These trends were also observed in the thermograms of the cellulose with the sorbed xylan as described above. These facts suggest that the interaction between cellulose and xylan in the blends is similar to that in the cellulose with the sorbed xylan. Based on these findings it is presumed that the volatile products from the decomposition of the xylan may be closely related to the interaction, because gaseous compounds can easily diffuse into the cellulose fibres.

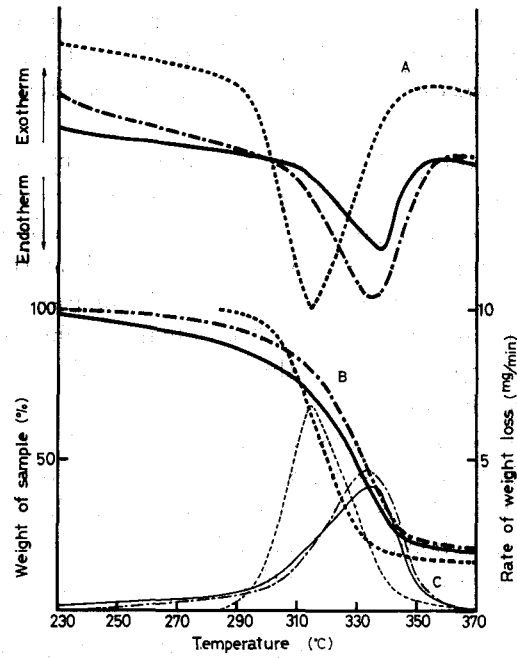


Fig. 5. Thermograms of blended samples in vacuum
 -----: C-0, - - - - -: Cellulose containing xylan (6%),
 ———: Cellulose containing xylan (12%)

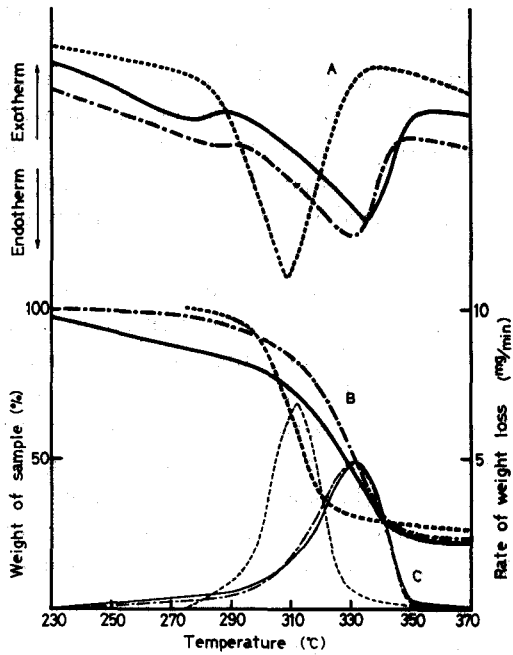


Fig. 6. Thermograms of blended samples in nitrogen stream
 legend: see Fig. 5.

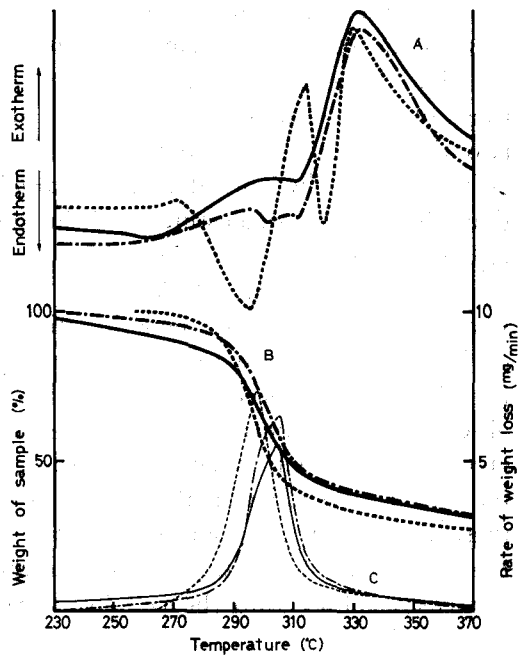


Fig. 7. Thermograms of blended samples in air
 legend: see Fig. 5.

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Summary

The thermal behavior of cotton cellulose containing the sorbed xylan was investigated by means of TG and DTA to study the effect of sorbed xylan on the thermal degradation of cellulose.

The cellulose samples of different quantities of sorbed xylan were prepared and their thermal analyses were carried out in vacuum, in a nitrogen stream and in air. The results showed that the initial stage of the thermal degradation of cellulose was affected by the presence of xylan. Taking into consideration the proposed mechanism of the thermal degradation of cellulose, the mechanism of interaction between cellulose and xylan was discussed.

The thermal behavior of physical blends of xylan and cellulose was also followed by TG and DTA. The TG curves obtained exhibited slightly different patterns from that of cotton cellulose containing sorbed xylan. However, the DTA curves showed trends similar to those of the samples of the sorption system.

On the basis of these facts, it was considered that the interaction between cellulose and xylan was caused by volatile compounds from the decomposition of xylan.

摘 要

キシランを吸着したセルロースの熱的性質を、TG および DTA で検索し、キシランのセルロース熱分解に及ぼす影響を検討した。

キシラン吸着量の異なる三種類のセルロース試料を調製し、それらの DTA および TG を真空、窒素気流および空気中で行った。その結果、セルロースの熱分解の開始温度は吸着キシラン量が増大するとともに、低温側に移動するが、分解速度は緩やかで、その最大重量減少速度の温度は高温側に移動する。また真空および窒素気流下での DTA は、重量減少とともに吸熱反応を示すが、そのピークは吸着キシラン量の増大とともに、高温側に移動し、ピーク面積は減少する。また空気中の DTA では、その分解初期の吸熱反応はキシランの存在することにより、発熱反応となる。これらの事実から、キシランの存在によって、セルロースの初期分解過程が影響されることが明らかとなった。また、これまで提案されたセルロースの熱分解機構を考慮して、熱分解時におけるキシランとセルロースの相互作用について、若干の考察を行った。

さらに、キシランとセルロースの単なる物理的混合試料を調製し、それらの熱的挙動を検索して、吸着系試料と比較検討した。その結果、TG 曲線は吸着系試料とやや異なった型を示したが、DTA 曲線は同様の傾向を示した。この事から、キシランとセルロース間の熱分解時における相互作用は、キシランの熱分解から生じる揮発性生成物によって引き起されるものと考えられた。