

## RESEARCH PAPER RP1326

Part of Journal of Research of the National Bureau of Standards, Volume 25,  
September 1940

## PECTIC SUBSTANCE OF COTTON FIBERS IN RELATION TO GROWTH

By Roy L. Whistler,<sup>1</sup> Albert R. Martin,<sup>1</sup> and Carl M. Conrad<sup>2</sup>

### ABSTRACT

The content of pectic substance in cotton fibers at various stages of growth was determined. The results suggest three distinct stages of development of the fiber. The first extends to about the 18th day and is characterized by a gradual decrease in the content of pectic substance. The second extends from the 18th to about the 35th day, during which time there occurs a rapid decrease in the content of pectic substance. In the final stage, which extends from about the 35th day to maturity, there is only a slight decrease in the content of pectic substance. These three stages of development are in good agreement with those deduced by other investigators from different types of data.

### CONTENTS

	Page
I. Introduction.....	305
II. Materials and methods.....	305
III. Experimental results and discussion.....	306
IV. References.....	308

## I. INTRODUCTION

It has been suggested [1]<sup>3</sup> that the pectic substance of cotton fibers is largely confined to the primary cell wall. Thus it would be expected that very young cotton fibers would contain a high percentage of pectic substance and that the percentage would decrease as fiber development progresses and would reach its lowest value at the time of fiber maturity. It was the purpose of the present investigation to examine this concept quantitatively by following the changes in percentage of pectic substance during the course of the development of cotton fibers.

## II. MATERIALS AND METHODS

The cotton fibers used in these experiments were collected during the summers of 1937 and 1939, from plants growing in the experiment station fields of the United States Department of Agriculture at Raleigh, N. C. The cotton was of the Mexican Big Boll variety of *Gossypium hirsutum*. The age of the fiber as collected in the field was determined by tagging the flowers on the day of blooming and

<sup>1</sup> Research Associates at the National Bureau of Standards, representing the Textile Foundation.

<sup>2</sup> Senior Cotton Technologist, Agricultural Marketing Service, United States Department of Agriculture.

<sup>3</sup> Figures in brackets indicate the literature references at the end of this paper.

then collecting the bolls at definite intervals. As soon as the bolls were picked, they were opened and the cotton fibers removed. The fibers which were to be used for the determination of uronic acids were quickly dried in a stream of warm air. Those used for the determination of pectic substance by the calcium pectate method were not dried but were quickly removed from the bolls at the time of picking and preserved in 80-percent ethanol.

Uronic acid determinations were made according to the procedure developed by Whistler, Martin, and Harris [2], and the results expressed as percentage of uronic acid-carbon dioxide on the basis of dry weight of the fibers, or as percentage of pectic substance. Conversion of uronic acid-carbon dioxide to pectic substance was accomplished by multiplying the percentage of carbon dioxide by the factor 4.8 [2]. The size of the samples taken for analysis varied from 2 to 10 g, depending upon the uronic acid content.

In addition to the determination of pectic substance by uronic acid analysis applied directly to the fibers, the 1937 series of fibers were analyzed for pectic substance by means of the calcium pectate method according to the following procedure. The sample (preserved in ethanol) was extracted 4 hours with 90-percent ethanol in a Soxhlet apparatus. The fibers, after drying in a vacuum oven at 60° C, were subjected to a 30-minute extraction with 100 ml of a boiling 0.5-percent solution of ammonium citrate, followed by three successive 30-minute extractions with fresh 100-ml portions of boiling 0.01 *N* hydrochloric acid. The entire cycle of four extractions was then repeated and the filtrates and washings were combined.<sup>4</sup>

The combined extracts were cooled, neutralized with sodium hydroxide solution, and the pectic substance was determined in an aliquot according to the procedure of Carre and Haynes [5]. The final calcium pectate precipitate was corrected for impurities by the method of Appleman and Conrad [6].

### III. EXPERIMENTAL RESULTS AND DISCUSSION

The pectic substance content in cotton fibers at various stages of growth is shown in table 1 and figure 1. It is seen that the percentage of pectic substance as determined by the calcium pectate method is consistently lower than that found by the direct method of uronic acid analysis. However, since methods of isolation in general give somewhat low results, this difference is not unexpected.

An examination of the curves in figure 1 reveals that they are composed of three major segments; the first, extending to about the 18th day, indicates a gradual decrease in the percentage of pectic substance with age of the fiber; the second, extending from about the 18th to the 35th day, indicates a rapid decline in the percentage of pectic substance; the third, extending from about the 35th day to the time of maturity, indicates only a slight decrease in the percentage of pectic substance with age of the fiber. It seems probable that each of these periods may be related to particular phases of growth in the development of the cotton fiber. For example, if it is assumed that the pectic substance is largely confined to the primary cell wall, no large change in the percentage of the pectic substance should occur

<sup>4</sup> The frequent change of solvent and the low concentration of hydrochloric acid were employed [3, 4] to minimize destruction of the pectic substance.

during the period of primary wall formation. However, as nonpectic material, for example cellulose, is deposited in the fiber, the percentage of pectic substance should decrease in approximate inverse proportion with the amount of deposited nonpectic substance.

TABLE 1.—*Calcium pectate and pectic substance content of cotton fibers at various stages of growth*

Age of fiber	Calcium pectate	1937 series		1939 series	
		Uronic acid-carbon dioxide	Pectic substance content (calc.)	Uronic acid-carbon dioxide	Pectic substance content (calc.)
Days	Percent	Percent	Percent	Percent	Percent
10	4.39	1.24	5.95		
11	4.02				
12	4.05				
14	3.95				
16	3.24				
17	3.24	1.16	5.57		
18	3.21	1.13	5.43		
19	3.58	1.01	4.85		
20	3.44				
21	3.12				
22				1.04	4.99
23	2.39	.73	3.50		
24				.79	3.79
25	1.85	.68	3.22	.60	2.88
26				.47	2.26
28					
30	1.29	.39	1.87	.35	1.68
32				.31	1.49
34				.24	1.15
35	.83	.30	1.44		
36				.23	1.10
39				.24	1.15
40	.62	.19	.91		
42				.18	.86
45	.79	.20	.96	.21	1.00
48					
50	.67	.22	1.06		
51				.22	1.05
54				.21	1.00
O.B.		.17	.82	.19	.91

It has long been recognized that the first phase in the growth of the cotton fiber consists in the development and elongation of the primary cell wall. The time required for the completion of this phase, as measured by Anderson and Kerr [1] for cotton fibers of the Mexican Big Boll variety, is about 20 days. This is very near the age at which the curves in figure 1 undergo a sudden and rapid decline. Thus the present data suggest that no major changes in the composition of the fiber occur during this phase of development.

With the completion of the development of the primary cell wall, the formation of the secondary wall is initiated and a rapid deposition of cellulose commences. This should result in lowering the percentage of pectic substance in the fiber, especially if no appreciable amounts of pectic substance are laid down with the cellulose. The second segment of the curve confirms such an interpretation and further indicates the rate at which the development of the secondary wall proceeds. This interpretation is also in accord with the work of Berkley [7], in which it was shown that in cotton fibers similar to those used in this work, the secondary cell wall underwent a rapid thickening between the 18th and 35th days of growth. An examina-

tion of the change in the percentage of cellulose with the age of the fiber further substantiates these views.<sup>5</sup>

From about the 35th day until the boll opened, the content of pectic substance underwent little change, as indicated by the third segment of the curve. This is in agreement with the findings of Berkley [7], who also showed that no significant change in the ratio of thin- to thick-walled fibers occurred during this period—a fact which indicates that no appreciable change in wall thickness is occurring.

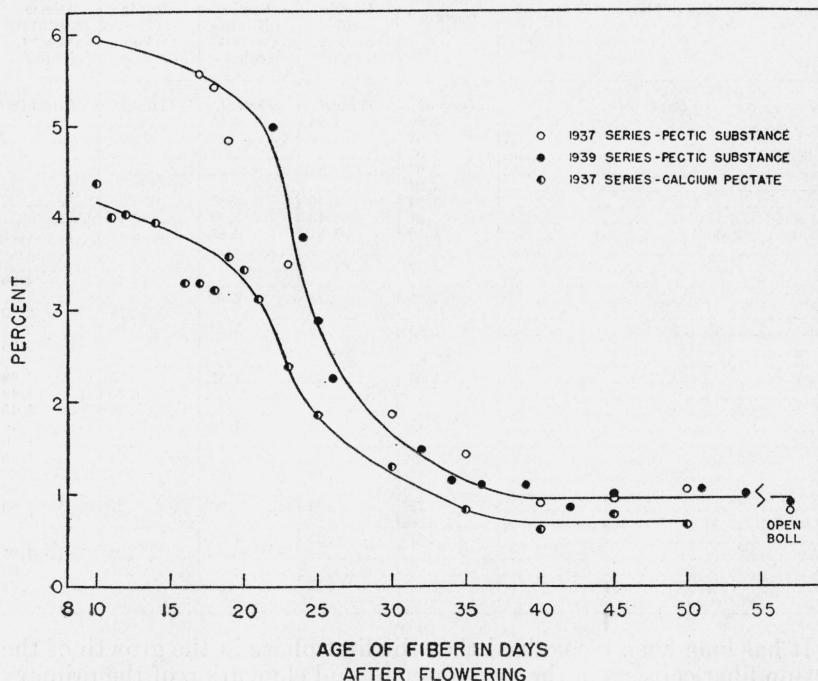


FIGURE 1. *The content of pectic substance of cotton fibers at various stages of growth.*

The upper curve was obtained from uronic acid analysis; the lower curve by isolation of the pectic substance as its calcium salt.

#### IV. REFERENCES

- [1] Anderson, D. B., and Kerr, T., *Ind. Eng. Chem.* **30**, 48 (1938).
- [2] Whistler, R. L., Martin, A. R., and Harris, M., *J. Research NBS* **24**, 13 (1940).
- [3] Conrad, C. M., *Am. J. Botany* **13**, 531 (1936).
- [4] Nightingale, G. T., Addoms, R. M., and Blake, M. H., *Bul. 494 N. J. Agr. Expt. Sta.* (1930).
- [5] Carre, M. H., and Haynes, D., *Biochem. J.* **16**, 60 (1922).
- [6] Appleman, C. O., and Conrad, C. M., *Bul. 291 Univ. of Md. Expt. Sta.* (1937).
- [7] Berkley, E. E., *Textile Research* **9**, 355 (1939).

WASHINGTON, May 29, 1940.

<sup>5</sup> This report is in preparation for publication.