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SCALE SUBSTANCE OF WOOL

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ABSTRACT

Earlier work at this Bureau has shown that wool that has been reduced with thioglycolic acid and then alkylated with ethyl bromide is attacked by pepsin in such a way that the scale material remains intact, whereas the interior of the fiber is completely dissolved. The composition of the scale material so obtained has now been studied. It has been found that it is essentially protein in chemical nature and, although it contains the same amino acids as the whole wool, the proportions of these in the two materials differ; thus, the whole wool used in this work contained 12.2 percent of cystine, 8.6 of arginine, 6.1 of tyrosine, and 9.5 of serine, whereas the scale material contained 20.3, 4.8, 3.3, and 11.2 percent, respectively.

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I. INTRODUCTION

A wool fiber usually consists of three concentric regions: (1) a thin outer layer called the cuticle, usually about 1 to 2 microns thick and made up of overlapping scales, (2) the cortex, or main body, of the fiber, and (3) a central pith, or medulla [1].[2] In fine wool, the last is generally lacking. The cuticle has been suggested to have a protective function, since it is mechanically tougher and chemically more stable than the rest of the fiber [1, 2, 3, 4]. It has been suggested also that the ability of wool fibers to felt is at least partially dependent on these scales (literature reviewed in [5]).

Little is known about the composition of the scales, since ready means for separating them from the rest of the fiber have not been available. From experiments involving partial hydrolysis of wool with hydrochloric acid, Trotman, Trotman, and Sutton [4] concluded that the scales were richer in sulfur than the cortex. Also, a number of investigators [6, 7, 8, 9] have concluded from color tests made under the microscope that the cuticle contains little or no tyrosine.

Recent work at this Bureau [10] has shown that wools chemically modified by reduction with thioglycolic acid, followed by alkylation

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[2] Figures in brackets indicate the literature references at the end of this paper.

with methyl iodide or ethyl bromide, are attacked by pepsin, and that the pepsin digests the inner portions of the fiber, leaving the cuticle intact. The present paper reports some analytical results on the composition of scale material obtained in this way.

II. EXPERIMENTAL PROCEDURE

1. REDUCTION AND ALKYLATION

The reduction and alkylation were carried out by methods similar to those previously described [11, 12]. A 140-g sample of wool was reduced for 24 hr at 35° C with 4,000 ml of a 0.2 *N* solution of sodium thioglycolate brought to pH 8.0 with potassium hydroxide. The yarn was then removed from the solution, washed thoroughly with water, and alkylated by treating it for 2 hr with 2,000 ml of a 0.1 *N* phosphate buffer solution at pH 8.0, in which had been suspended 25 g of ethyl bromide. After again washing the wool with water, the processes of reduction and alkylation were repeated. The product was again washed, dried, and finally comminuted in a Wiley mill.

2. TREATMENT WITH PEPSIN

The chemically modified wool was suspended in 3,000 ml of a 0.2 *M* solution of potassium chloride, which was adjusted to pH 1.1 by the addition of hydrochloric acid, and which contained 1.0 g of pepsin.[3] The suspension was kept at 35° C and slowly stirred for 5 days. After this length of time, examination under the microscope indicated that the residue consisted almost entirely of scales, and that all the cortical material had been dissolved.

The scale material was separated by centrifuging and was washed several times with a 5-percent solution of potassium chloride and several times with distilled water. The material then was dried by washing it with alcohol and then with ether, and the final drying was done over concentrated sulfuric acid in a vacuum desiccator. The dried product formed a light-brown, horny mass weighing 3.283 g, representing about 2.3 percent of the weight of the wool.

III. RESULTS AND DISCUSSION

Preliminary qualitative tests of the scale material indicated that it was composed largely of protein. It gave positive xanthroproteic [13] and biuret [13] reactions. Moreover, the Millon [13] reaction, given by proteins containing tyrosine or tryptophane, the alkali-plumbite [14] reaction, given by those containing sulfur, and the Sakaguchi [13] reaction, given by those containing arginine, were all positive. The Ehrlich [13] reaction for amino sugars was negative. Only a faintly positive test for carbohydrate was obtained when the Molisch [13] test was applied. No carbohydrate could be detected by using Fehling's solution or by Beek's [15] method. A test for bound lipid by the method of Anderson [16] showed that the material contained 2.7 percent of lipid. On ignition [17], 4.13 percent of ash was found to be present.

Determination of nitrogen by the micro-Kjeldahl procedure of Clarke [18] gave 13.53 percent, somewhat less than the amount usually

[3] From Fairchild Bros. & Foster, New York, N. Y.

found in proteins, but correction for ash, lipid, and the ethyl groups introduced chemically increased this to 15.17 percent, a value just slightly lower than that given by wool (table 1). This difference seems largely due to the smaller arginine content of the scale material, which contained only about half as much arginine (4.8 percent) as whole wool (8.6 percent). Arginine is the richest in nitrogen of the amino acids. The scale material contained 5.42 percent of sulfur [20], which is considerably more than that in untreated wool (3.50 percent).

TABLE 1.—*Composition of untreated wool and of wool scales*

Constituent	Untreated wool	Wool scales	
		Found	Corrected value [a]
Sulfur	3.50	4.83	5.42
Cystine	12.2	[b]18.1	[b]20.3
Nitrogen	16.67	13.53	15.17
Arginine	8.6	4.3	4.8
Tyrosine	6.1	3.0	3.3
Serine	9.5	9.9	11.2
Ethyl groups	0.0	4.0	---
Ash	.2	4.1	---
Lipid	---	2.7	---

[a] Corrected for the presence of ethyl groups, ash, and bound lipid, as described in the text.
[b] Calculated from the sulfur content.

The sulfur in the scale material probably was largely in the form of S-ethyl cysteine, since the determination of the ethyl groups [21] showed that 4.0 percent was present, a result which indicates that 1.51 millimoles of sulfur and 1.38 millimoles of ethyl groups were present per gram of scale material. This S-ethyl cysteine had doubtless been formed from cystine by the reduction and alkylation processes used in preparing the material.

Determination of the tyrosine in the scale material [22] showed that it contained only about half as much (2.97 percent) as wool (6.10 percent). Analysis by the method of Nicolet and Shinn [23] showed that the scale material contained about the same amount of serine as wool.

IV. CONCLUSIONS

Analytical studies of the cuticle of wool and of whole wool fibers show that although both contain the same amino acids, the proportions of these in the two materials differ. The presence of larger amounts of sulfur presumably means that the protein of the scales contains more sulfur cross-links between its peptide chains than do the more digestible proteins of the cortex. Nevertheless, even when the sulfur cross-links have been broken as in the present product, the material is not digested by enzymes. Moreover, the scale material was found to be more stable toward alkali than wool that had been reduced and alkylated in the same way, since the alkali-solubility [24] of the scale material was found to be only 42 percent and that of the treated wool 85 percent.

Two possible explanations have been advanced to account for the difference in behavior of the cuticle and the cortex of wool. Speakman, and also Rudall, have suggested that, since the cortex of wool

fibers is attacked more rapidly by sodium sulfide than are the scales, cross-links other than those involving sulfur may be present. This hypothesis is supported by the results of the present work, although direct proof of the existence and nature of such cross-links is still lacking. An alternative explanation might be sought in the recent demonstration by Hock and McMurdie [25], with the electronmicroscope, that the cuticle and cortex differ widely in physical organization.

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