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THE EFFECT OF ULTRA-VIOLET RAYS UPON THE BREAKING STRENGTH OF WEIGHTED AND UNWEIGHTED SILK FABRICS

by

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INTRODUCTION

Because the consumer is interested in obtaining from a fabric the maximum of service for the price paid, the manufacturer is always confronted with the problem of determining the factors which cause the deterioration of the various fibers. Sunlight, moisture, temperature, perspiration, dyes, and friction have been found to cause a change in the wearing qualities of a fabric, but the degree to which each influences the strength of the fabric is a problem by no means entirely solved.

The effect of light on silk may easily be overlooked by most individuals, but it is undoubtedly an important factor to consider, especially in some localities. The relative effect of light on the durability of weighted as compared with that on unweighted silk has in recent years claimed the attention of several investigators, but many questions are still unanswered.

Studies of the nature and action of ultra-violet rays within recent years have made it seem probable that the changes brought about in fabrics by sunlight are largely due to these rays. The development of mechanical devices to produce the ultra-violet rays artificially have made it possible to make much more accurate studies than formerly. The ultra-violet from artificial sources can be produced in

much greater intensity than that from the sun and conditions can be more satisfactorily controlled; consequently, in most experimental work the ultra-violet from artificial sources is used.

The purpose of this study was to determine if possible the effect of ultra-violet rays on weighted and unweighted silk. The effect of light upon different textile fibers has been studied in a number of laboratories throughout the country, and it is hoped that the results from all of the experimental work may aid the manufacturer in formulating quick and satisfactory methods for testing the influence of light upon his product. If definite tests can be developed, the consumer may be very decidedly benefitted.

REVIEW OF LITERATURE

Although exposure to sunlight had long been recognized as one of the factors influencing the deterioration of fabrics but little scientific study of the problem was made until the World War. The growing importance of airplanes in warfare at that time made it necessary to study the causes for the deterioration of the fabrics used in their construction and to devise means for protecting the fabrics (1). In his study of factors causing the deterioration of airplane fabrics, Turner (2) found light to be the most important one. The British Aeronautical Research Committee

(1) published information as to methods of excluding light from the fabric and of incorporating substances in it to resist the action of light.

Sir Isaac Newton gave the world its first accurate information concerning the nature of light in 1666. The simplicity of his experiment is fascinating and the same principle is still used in the study of light. He cut a small hole in a window shade, and held a prism so that a beam of light would strike the prism and be refracted. Newton saw the colors violet, indigo, blue, green, yellow, orange, and red, which he called the spectral colors. He saw only the visible colors and did not know that beyond the violet was the ultra-violet and beyond the red, the infra-red rays, the former too short and the latter too long to give to the human eye the sensation of color (3).

It was not until 1801 that the ultra-violet spectrum was discovered by Ritter. Because of the shortness of the wave length, the ultra-violet spectrum could not be described in terms of color; consequently, it has been divided according to the wave length of the rays (4).

The term ultra-violet is often used to designate a region of the invisible spectrum, which is rather indefinite, but is usually considered as the protion having wave lengths below 400 millimicrons (mu), or 4,000 Angstrom units (A°). The visible spectrum lies between the region of 4,000 A°

and 7,000 A°. The infra-red extends from 7,000 A° to an indefinite region beyond 30,000 A° (5).

Although the term is not entirely accurate the expression ultra-violet is usually used to include all rays within the ultra-violet spectrum. In the visible spectrum, the waves producing red are specifically distinguished from those producing orange, while in the ultra-violet spectrum, rays of entirely different lengths are all designated by the same term, ultra-violet. When more is known about the region of the ultra-violet more correct terms may be used (4).

"Natural radiation commonly called daylight consists of, (1) direct solar radiation, (2) diffuse radiation from the sky, and (3) radiation reflected from surroundings such as trees, buildings, etc." (3). It is evident that radiation from natural sources may vary during different hours of the day, at different seasons, and in certain geographical localities. Results of the work of Dorno (6), in Davos, Switzerland, showed the extremes of the ultra-violet for July 15 to be 200 arbitrary units before 7:00 A. M. and 1150 units shortly before noon. For January 15, the extremes were 100 units at 10 A. M. and 200 units shortly after noon. He emphasized the fact that the variation in natural radiation should be considered in using artificial radiation with the intention of making comparisons between the two.

Pycha (7) measured the ultra-violet radiation at Man-

hattan, Kansas every day for one year. He found the radiation for January 15 to be 2 units and for July 15, 16 units. This variation in radiation at extremes of the year is in agreement with the data from Dorno's investigation.

That the temperature was unimportant was shown by the decreased readings for the month of October, although some of the days in October were warmer than days in the summer when the readings were highter. In his work with the mercury arc lamp, Pycha obtained the same unit with exposures made at temperatures of 0°C, 20°C, and 37°C.

Authorities have disagreed as to the exact course for the absence of some of the ultra-violet rays in the lower atmosphere. Luckiesh (3), considered as one of the best authorities on light, has published valuable information on the subject. He reports that radiation for a certain hour of the day depends upon the altitude and condition of the atmosphere. The presence of moisture, dust, and smoke in the air reduces the transmission of light rays through the atmosphere to the earth.

A number of investigators, in an attempt to confirm their belief that the limit of the solar spectrum is due to ozone, have furnished strong evidence but the thing has not been definitely proved.

Fabry and Buisson (5) showed rather conclusively that in the upper atmosphere the oxygen is converted into ozone

by the rays in the extreme region of the ultra-violet spectrum. The ozone then absorbs the radiation near 3,000 A° and allows only one part in one hundred thousand to pass through at 2,920 A°. If the radiant energy reached the surface of the earth without any alteration due to the atmosphere, it would be approximately fifty times stronger than it actually is under the most favorable conditions.

Kreusler (3) found that in a column of air 20.45 centimeters long at 747 millimeters pressure, and 14°C temperature, there was an absorption of 8.8 per cent of the radiation at 1,869 A° and none beyond 1,930 A°. In studying the extreme region of the ultra-violet spectrum it is necessary to use vacuum spectroscopes.

Ultra-violet radiation is absorbed to a considerable degree by reflection, each substance having a definite reflection-factor. Many substances reflect the rear ultra-violet but will absorb the radiation from the middle and extreme regions. Luckiesh (3) cites snow as an example of an excellent reflector of the ultra-violet as well as of the visible radiation. In photo-chemical activities where it is necessary to reflect at least the near ultra-violet, consideration of the reflection factors of substances is essential. Not only the color, but also the kind of surface, the substances, and combinations must be considered, for so-called "white" substances may have very different

reflection-factors. It is necessary to determine the reflection-factor of a substance if one is to know accurately the rays which are absorbed and those which are reflected.

Doree (8) concluded from his work that the deterioration of fabrics upon exposure to sunlight was due to the ultra-violet radiation. The rays may act directly upon the fabric causing it to lose strength or they may form ozone which then acts upon the fibers. Heermann (1), who has done some of the most thorough work with ultra-violet and its effect on fibers, concluded that deterioration was due to the action of the ultra-violet rays. However, other authorities believe the loss in strength of the fabrics is due to the action of the ozone in the presence of moisture.

The results of Barr's (9) work on the action of light on textiles show coarse yarns to be less affected by the light than fine ones; the outer layers in the coarse yarns serve as a protection to the inner layers. The weakening of cotton and linen fibers seemed to be caused by rays less than 4,000 A°. In other words, the ultra-violet radiation had a much greater effect on the fibers than the visible radiation.

In his work with silk and linen, Vignon (10) exposed pieces of the fabrics to sunlight, dry heat, moist heat, and moisture with and without sunlight. The silk material resisted the action of moisture in the absence of sunlight

better than in the presence of the light. One might conclude from his report that deterioration of silk material upon exposure to sunlight is greater when the silk is moist than when it is dry.

From the results of Urquhart's and Williams' (11) investigation on the absorption of moisture by textile fabrics, it seems that materials of the same fiber and construction may have different moisture regains. They state that for a given humidity there are several possible values of moisture content, this depending upon the previous history of the material examined. A study of fabrics showed the moisture content at a constant relative humidity to decrease with an increasing temperature, but the decrease became less marked the nearer the humidity approached the point of saturation.

For many years chemists thought the loss in strength of silks upon exposure to light was due entirely to metallic weighting. Heermann (1) disproved this theory. He showed that silk in any form whether raw, degummed, or weighted was more sensitive to light than any of the other fibers. He found that raw silk placed sixteen centimeters from a 1,600 power mercury arc lamp lost 50 per cent of its strength after six hours exposure; degummed silk lost the same amount of strength in from two to two and one-half hours; and weighted silk, depending upon the amount of weighting, lost

50 per cent of its strength in from one to one and one-half hours exposure. From information secured on weighting it would appear that loss in strength of weighted silk upon exposure to light might vary a great deal.

Of all the work done on silk weighting, that of Stockhausen (12) has been outstanding. He states that a tin and tin-phosphate weighted silk is more greatly injured by radiation than a tin-phosphate-silicate weighted silk, the crystalline structure of the stannic acid playing an important part in weakening the silk fiber. Consequently, the kind of weighting used in the silk may make a difference in its resistance to light. Because of the lack of knowledge on the subject, Stockhausen found it very difficult to summarize results or make general statements concerning the change in tensile strength of silks upon exposure to light.

The changes which take place when a textile fiber is exposed to ultra-violet rays depend upon the physical and chemical structure of the fiber (13).

Raw silk consists of two fine transparent filaments of fibroin cemented together by sericin. The fibroin constitutes about one-half to two-thirds of the raw silk. Most of the remaining weight is due to sericin although there may be small quantities of fats, waxes, and mineral matter, which are removed in the degrumming process. The degummed silk is pure fibroin.

The exact composition of the silk protein, fibroin, has not been determined. Analyses made by Mulder, Vignon, and other (14) give fibroin as consisting of the four elements, carbon, hydrogen, oxygen, and nitrogen. Morel and Sisley (15) found that the protein contained traces of sulfur also.

PREPARATION, ANALYSIS, AND TREATMENT OF MATERIALS

Apparatus Used

A Standard Luxor Model Alpine Sun Lamp was used as the source of light. It had a quartz mercury arc adjustable to various heights.

To make the results of this study of value in comparing with other work simple physical analyses of the silk materials were necessary.

The thread count, or the number of threads per inch, was made with a Lowinson's micrometer for counting threads in fabrics. The number of threads per inch was used as a basis for the count. Due to the regularity in number of picks and ends, the thread count of the specimens was based upon the average count of ten warp and ten filling strips.

The thickness of the materials was measured with a Randall and Stickney thickness gauge. The average of ten measurements was taken as the thickness. The methods for

measurement of thickness and the thread count were carried out according to test methods recommended by the American Society for Testing Materials (16). The results of these tests are shown in Table I.

Table I. Analysis of Silk Fabrics

Name of Material	Weave	Width	Thickness	Thread Count ends	Thread Count picks	Weighting in per cent
Silk Crepe (A)	Plain	36 In.	.0122 in.	184.2	87.8	00.0
Silk crepe (B)	Plain	36½ "	.0097 "	171.0	85.1	64.0
Silk crepe (C)	Plain	36¼ "	.0120 "	179.6	87.3	87.0

All breaking strength tests were made with a Combination Scott Tester which was installed in the conditioning room. A temperature of 78°-83°C and a relative humidity of 60-65 per cent were maintained in the room by a Carrier unit air conditioner.

Preparation of Silks

The silk materials used were undyed crepes prepared under controlled conditions in the research laboratory of Cheney Brothers. The gum was removed from a piece of the silk fabric by immersing it for two hours in an ordinary

soap solution at 96°-98.7°C. It was rinsed first in a dilute ammonia solution and then in warm water and air dried. The degumming process was as mild as could be used to remove the sericin satisfactorily. Although some change in the nature of the protein of the silk may have taken place, there was no greater reaction than would occur ordinarily in the processing of silk.

The piece of degummed silk was used as the unweighted silk in this experiment. Two pieces of the degummed silk were weighted with different amounts of weighting. They were used as the weighted silks.

A tin-phosphate silicate weighting was used in preparing the weighted silks. An analysis of the materials showed one, material B, to have 64 per cent of weighting and the other, material C, to have 87 per cent of weighting.

The Bureau of Standards method of calculating the percentage weighting was followed (17). The percentage total weighting in the silk fabric is based upon the weight of the pure silk fibroin. In order to determine the weight of the silk fibroin a sample of the weighted silk was dried and weighted. The weight was called A. It was then treated with water to remove what is termed the finishing materials and weighted again, giving weight B. A treatment with hydrofluoric-hydrochloric acid (H_2F_2-HCL) reagent removed the metallic weighting. The weight of the sample at this stage

was called C. The sample was ignited at high temperature to remove the small amount of carbonaceous material present. The final weight of the ash was D. The formula worked out for calculating the total percentage weighting is:

$$\frac{A-C+D}{A} \times 100 = \text{per cent of weighting}$$

Preparation of Specimens

The silk specimens were prepared by the strip method according to specifications recommended by the American Society for Testing Materials (16). Each specimen was cut six inches long and one and one-fourth inches wide and raveled to a width of one inch. A micrometer was used to get the exact width in each case. No specimens were cut nearer to the selvage than one-tenth of the width of the fabric (18).

The prepared specimens were divided into sets, a set consisting of five warp and five filling strips. The sets were numbered I, II, III, IV, and V, according to the condition under which each was treated. The letter A was used to designate the unweighted silk; B, the piece containing 64 per cent of weighting; and C, the one containing 87 per cent of weighting.

Each set was placed between single thicknesses of filter paper and kept in the darkened conditioning room except during the time of exposure to the ultra-violet radiation.

METHOD OF PROCEDURE

In a study of this kind, the length of rays, the source of light, and the time of exposure are some of the phases that might be considered. It was thought best to confine this study largely to the time of exposure. Some work was done on other phases, but not enough to make definite conclusions concerning the results of that part of the work possible.

In considering the length of time of the exposures it was believed that data on the effects produced by a short exposure would be of more value than from a long exposure. It would be possible to control conditions to a better advantage if the exposure was short. While most of the exposures were of relatively short duration some specimens were exposed for eight, sixteen, and thirty-two hours.

Probably the greatest possibility for error in the experimental work was in the exposure of the silks to the ultra-violet radiation. In order to control conditions as nearly as possible in exposing the silk, the current was turned on at least ten minutes before the specimens were placed under the lamp. The specimens were laid on the filter paper in which they were kept and then placed on a corrugated paste board covered with two layers of white cotton toweling. A position was marked on the table where exposures

were made so that each time the light and specimens might be placed in the same position as that used in previous exposures. The arc was thirty inches from the specimens. Whenever more than one exposure was made the position of the sets of specimens was alternated upon exposure so that the radiation might be more nearly the same for each set of specimens in each condition. This was done because it was thought that there might be a variation in the radiation over the exposed surface. Tests on the lamp showed that there was some variation in the radiation.

For the short time exposures, in which all specimens were exposed air dry, five sets of each material were placed in the conditioning room at the same time. Each set was folded between a single thickness of filter paper and placed on a table in the center of the room. One set (1) was used as a control. The breaking strength of the control was taken as the basis for calculating the loss in strength due to exposure to ultra-violet rays. Set (II) was exposed for one thirty minute period; set (III) for two thirty minute periods; set (IV) for three thirty minute periods; and set (V) for one sixty minute period. The work was planned so there would be a lapse of forty-eight hours between exposures and the exposures would all be completed at the same time. This made it possible to test the breaking strength of the five sets of each material at one time. The speci-

mens were folded in filter paper and kept in the conditioning room between the periods of exposure.

Specimens which were subjected to the light for thirty-two hours were exposed in two hour periods with a lapse of twenty-four hours between exposures. Those which were treated for sixteen hours were exposed in two hour periods with forty-eight hours between. In the sixteen and thirty-two hour exposures the strips were air-dry. The procedure for exposing the strips was the same in all cases.

In comparing the effect of ultra-violet on wet and on dry silk material, an eight hour exposure was made in two hour periods with forty-eight hour intervals between exposures. The specimens were saturated with distilled water just before each exposure, but they became completely dry soon after they were placed under the lamp. All specimens were conditioned after the last exposure for a period of from six to twenty-four hours before being broken.

The tests were made by the strip method. The jaws of the Scott Tester were adjusted three inches apart. Each specimen was clamped in the jaws so that the pull would be uniform and the specimens broken accurately. The breaking strength was recorded for each specimen, and in part of the work, both stretch and strength were recorded on the recorder of the machine.

The wide differences in the breaking strength of the

specimens after exposure led to the belief that the radiation from the lamp must have been irregular. There was no satisfactory means of making an absolute determination of the intensity of the radiation, but a relative determination, which indicated the changes in intensity, was made by the method worked out by the Hanovia Research Laboratory (19), makers of the Alpine sun lamp. It was carried out as follows:

Equal amounts of oxalic acid-uranyl sulfate solution were measured into quartz test tubes, stoppered with corks and exposed to the ultra-violet radiation at a distance of thirty inches from the arc. After ten minutes of exposure the solution was titrated with N/10 potassium permanganate ($KMnO_4$). The test tubes were placed in the field of radiation in the same positions as the specimens of silk had been placed. Nine sets of tests, each in duplicate, were made. The $KMnO_4$ was standardized each time it was used.

Table II. Effect of Short Exposures to Ultra-Violet

(a) Average Pounds Breaking Strength

Material	Control		30 Min. exposure		30-30 min. exposure		30-30-30 min. exposure		60 min. exposure	
	Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling
A	50.7	25.8	50.7	25.4	50.0	25.6	51.4	25.9	51.0	25.1
B	48.2	17.3	47.6	17.2	46.0	17.3	46.7	17.3	47.9	16.7
C	46.5	15.4	45.9	15.1	44.4	15.4	44.5	15.5	45.7	15.7

(b) Percentage Loss in Breaking Strength

Material	30 min. exposure		30-30 min. exposure		30-30-30 min. exposure		60 min. exposure	
	Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling
A	0.00	1.55	1.38	0.77	+1.38	+.39	+.59	2.71
B	1.24	0.57	4.56	0.00	3.11	0.00	0.62	3.46
C	1.72	2.59	4.52	0.00	4.30	+.64	1.72	+1.94

Table III. Effect of Long Exposures to Ultra-Violet

(a) Average Pounds Breaking Strength

Material	Control		8 hrs. exposure		Control exposure		16 hrs. exposure		32 Hrs. exposure	
	Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling
A	47.4	20.9	49.0	22.4	44.0	23.1	37.0	18.6	35.5	15.5
B	45.5	15.4	44.1	14.9	42.3	15.9	37.0	12.1	31.1	12.8
C	43.7	13.4	41.8	13.6	43.5	14.6	37.0	10.6	33.6	10.2

(b) Percentage Loss in Breaking Strength

Material	8hrs. exposure		16 hrs. exposure		32 hrs. exposure	
	Warp	Filling	Warp	Filling	Warp	Filling
A	+3.37	+0.71	15.90	19.48	19.31	32.90
B	3.07	3.24	12.52	23.90	26.47	19.49
C	4.34	1.49	14.94	27.39	22.76	30.13

Table IV. Effect of Ultra-Violet in Presence of Moisture

(A) Average Pounds Breaking Strength

Material	Control		8 hrs. exposure (dry)		8 hrs. exposure (wet)	
	Warp	Filling	Warp	Filling	Warp	Filling
A	47.4	20.9	49.0	22.4	45.5	23.0
B	45.5	15.4	44.1	14.9	38.0	13.3
C	43.7	13.4	41.8	13.6	40.0	11.7

(b) Percentage Loss in Strength

Material	8 Hrs. exposure (dry)		8 hrs. exposure (wet)	
	Warp	Filling	Warp	Filling
A	+3.37	+0.71	4.00	+1.00
B	3.07	3.24	16.48	13.63
C	4.34	+1.46	8.46	12.68

DISCUSSION OF RESULTS

Studies made of the effect of ultra-violet on the tensile strength of textile fabrics show that silk is the most easily affected of all fibers (20). This is probably due to the physical and chemical structure of the fiber. Silk is the finest of all fibers and because of the fine filaments it is easily penetrated by light rays. Because only a limited study has been made of the chemical structure of silk, little is known about the action of light on the fiber.

Specimens exposed to the rays from eight to thirty-two hours were yellowed. This yellowing might indicate that an oxidation reaction had taken place for proteins become yellow as oxidation proceeds. The loss of strength and reduced elasticity of the silk may have been due to an oxidation process which was greatly accelerated by the action of rays from the lamp.

There was no available method for determining the extent of oxidation. If all the change in the fiber were the result of oxidation it would be possible to study the relative amount of the oxidation reaction by measuring the change in color with a colorimeter; the yellowing produced upon exposure to light would serve as an indication of the extent of the oxidation.

The data in Tables II-a and II-b show that in most cases the silk exposed for as short a period as one-half hour lost some strength. Repeated exposures for short periods apparently cause a greater deterioration than one continuous exposure for the same length of time. Nothing was found in the literature to substantiate such a theory, but it appears that if the reaction is one of oxidation the process might continue for an indefinite time after the exposure to the ultra-violet had ceased. If this were true, specimens exposed for two thirty minute periods should be oxidized to a greater extent than those exposed for one continuous sixty minute period, and the greater the amount of oxidation the greater the tendering of the fabric.

The chemical change in the silk upon exposure to the ultra-violet radiation is evidently influenced by the moisture content of the fabrics when first placed under the light. Tables IV-a and IV-b show that in most cases there was decidedly a greater deterioration in silks which were saturated with water before exposure than in the air-dry specimens. From the investigations of Cunliffe and Farrow (21) and of Vignon (10) it would seem that moisture in the presence of light might hasten the decomposition of the fabrics.

In most cases the weighted silks lost more strength than the unweighted silk. However, in a number of instances

material B, which contained the smaller percentage of weighting, lost as much or more strength than C, the more heavily weighted piece. Since the same kind of weighting was used in both, the variations could not be due to a difference in weighting material. Those results are not in accord with those of Heermann (1) for he claims that a more heavily weighted wilk should lose more strength than a less heavily weighted one.

This difference in strength after exposure might be explained by the degree of protection furnished by the metallic weighting. The larger amount of weighting in C may aid in preventing the ultra-violet light from penetrating the fiber, while in B. the quantity is insufficient to afford as much protection. In both cases the weighting evidently caused a greater loss in strength.

The results obtained were not always consistent. There may be several causes for such differences. In the first place, there was a wide variation in the breaking strength of the unexposed specimens used as controls. This was especially true of the unweighted silk.

Another cause for differences in strength was doubtless due to variations in the intensity of the ultra-violet radiation, caused by fluctuations of the current and the position of the specimens when exposed.

SUMMARY

1. The data indicates that silk fabrics, whether they are weighted or unweighted, are tendered by the action of ultra-violet radiation. The extent of the deterioration is greater in weighted silks, but it appears not to be in proportion to the amount of weighting in the fabric.

2. From the data obtained in this investigation the loss in tensile strength upon intermittent exposures appears to be greater than upon a continuous exposure for the same length of time.

3. There is also an indication of a greater deterioration of the silk fabrics when they are exposed to ultra-violet in the presence of moisture. The moisture seemingly hastens the rate of the chemical change.

4. For white silk the degree of yellowing may indicate the extent of the chemical change accelerated by the ultra-violet radiations.

The results obtained in this investigation show that more work must be done before definite conclusions regarding the action of ultra-violet upon the strength of silk fabrics may be made.

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