

THE EFFECT OF ULTRA-VIOLET RADIATION AND PERSPIRATION
ON THE BREAKING STRENGTH OF CERTAIN SILK FABRICS

by

ESTHER MARGARET CORMANY

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INTRODUCTION

The deterioration of silks has claimed the attention of a number of investigators in the last few years. Some silk fabrics are known to give poor service because of a tendency to decompose under the influence of such agencies as weighting, light, perspiration, mechanical abrasion, and reagents used in laundering and dry cleaning.

The purpose of this experiment was to determine, if possible, the effect of perspiration and ultra-violet radiation on the breaking strength of certain unweighted and weighted silk fabrics.

REVIEW OF LITERATURE

With the discovery of the spectrum Sir Isaac Newton in 1666 revealed an unexplored region pertaining to the composition of light and of radiant energy in general. He saw only the visible radiation--light--and gave to the spectral colors the names, violet, indigo, blue, green, yellow, orange and red. The ultra-violet and infra-red regions were discovered almost simultaneously. W. Herschel discovered infra-red in 1800; in 1801 Ritter noted the effect on silver chloride of what proved to be ultra-violet radiation (Luckfiesh, 1922).

Until the invention of the electric dynamo the only adequate source of ultra-violet radiation was the sun. The

sun is unsteady, uncertain, and discontinuous as a source and limited in spectral range in the ultra-violet region. The carbon arc was the first artificial source of appreciable power, and it may be said that the twentieth century was the first to have artificial sources of this energy sufficiently efficient and adequate to draw marked attention to applications on a commercial scale (Luckiesh, 1922).

Ultra-violet rays embody a range of wave length, just as light rays represent a range. Because of the shortness of the wave length, the ultra-violet spectrum cannot be described in terms of color, but has been divided according to the wave length of the rays. The ultra-violet spectrum is an invisible area having wave lengths below 400 millimicrons (μ), or 4000 Angstrom units (A°).

"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, and similar surfaces" (Luckiesh, 1922). It is known that radiation from natural sources varies with the time of day, with the season of the year, and in different sections of the country. In an article in the Industrial and Engineering Chemistry (1929), Dorno, concluded from his work that we should consider this variation in natural radiation when using artificial radiation, if we wish to make a comparison between the two.

Pycha (1928) found from his work that temperature had

no effect on radiation.

Coblentz (1929) and his associates state that natural silks when not yellowed with age have a high transmission of ultra-violet radiation. Also, the ultra-violet rays (and to a less extent the longer wave lengths) are greatly absorbed by natural silk that is yellowed with age.

According to Johnson (1927), investigations made by Heermann indicate that the deterioration of silk is the result of ultra-violet radiation rather than a secondary reaction due to the ozone produced during radiation. However, other authorities believe that ozone is developed and acts strongly on the fibers in the presence of moisture.

Doreo (1917) maintains that the deterioration is caused by ultra-violet radiation, acting alone or in conjunction with the ozone produced by the radiation.

Quoting from Harris and Jessup (1931), "K. Homolka and P. M. Grempe state that the weakening of silk fibers by radiation is caused by the short ultra-violet rays from the sun."

The results of Vignon's (1920) work would indicate that silk material is more affected when exposed to sunlight while moist than when exposed to sunlight while dry.

Heermann, according to Johnson (1927), disproved the commonly accepted theory that the loss in strength of silks when exposed to light was due to metallic weighting. He found that both unweighted and weighted silks were affected

by light, and that the loss in strength of the weighted silks depended upon the amount of weighting.

Stockhausen (1930) believes that the kind of weighting used, may make a difference in its resistance to light. He showed that a tin or tin-phosphate weighting in silks causes a greater loss when exposed to light than a tin-phosphate-silicate weighting. He believes crystalline structure of the stannic acid plays an important part in weakening the silk fiber.

Most investigators have limited their studies to the effect of ultra-violet radiation on the breaking strength of the silk, but the protein chemist had studied the chemical changes produced by the radiation. According to Harris and Jessup (1931), Effront has decomposed amino acids and protein matter in an alkaline solution with the formation of ammonia and volatile acids. He further found that sterile solutions of peptones exposed to sunlight decompose with the formation of peroxide, nitrates, ammonia, and volatile acids. He concluded that the active agent was hydrogen peroxide, as this will decompose peptones and amino acids.

Perspiration is a mixture of the liquids, sebum and sweat, each of which is secreted by a special gland. These two secretions differ widely in composition and quantity produced. The volume of sebum secreted is fairly constant. The amount of secretion from the sweat glands depends upon the temperature and humidity of the atmosphere, the tempera-

ture and condition of the body, the nature and quality of food ingested, and the presence or absence of chemicals which have a stimulating effect upon the glands. A high atmospheric temperature and a low relative humidity or muscular exertion favor production of perspiration (Trotman, 1929).

The reaction of perspiration may be slightly acid or slightly alkaline depending on the proportions of sebum and sweat. Sweat alone is normally alkaline, and sebum is acid. The average composition of perspiration may be taken as water 98 per cent, saline matter 0.6 per cent, organic matter 1.4 per cent. Saline matter consists chiefly of sodium chloride derived from sweat (Vass and McSwiney, 1929).

The action of perspiration on textiles is stated to be a combination of the effects of acidity or alkalinity, of the sodium chloride, and the results of bacterial growth (Vass, 1929).

Sodium chloride, which is always present in perspiration, is the principal cause of the tendering of silk by perspiration. There is not the slightest doubt that a very dilute solution of sodium chloride dried on silk causes gradual disintegration. It is not known how this occurs but Dreaper, according to Trotman (1929), suggested it might be due to the formation of hydrochloric acid by hydrolysis. Silk absorbs hydrochloric acid readily and this would prevent the hydrolysis represented by the equation

$\text{NaCl} + \text{HOH} \rightleftharpoons \text{HCl} + \text{NaOH}$ from establishing an equilibrium. Hydrochloric acid is oxidized by the oxygen with the formation of chlorine which would react with the protein of the silk to form chloramines. An alternative explanation may be that chlorine ions are liberated by dissociation and enter at once into combination with the silk, the sodium ions combining with the carboxyl group (Trotman, 1929).

When a fabric is moistened with perspiration it becomes an ideal medium for bacteria. These develop rapidly and produce further destruction of the epithelial tissue and of the proteins, but it is hard to say without comparison with the original fabric how much is due to bacterial action. Johnson (1927) refers to an article published in the Journal of the Society of Chemical Industry which claims that although the silk fibers may be discolored they are not tendered by bacteria.

The weighting of silk will probably never be abandoned by silk manufacturers, and unless carried to extremes, it need not be harmful to the silk. Overweighting has an injurious effect upon the breaking strength and elasticity of the fabric, due to a partial disintegration of the fiber (Johnson, 1927).

A large percentage of the formulae for weighting silk include some form of tin. Salts of tin which are most commonly used are stannous chloride, stannic chloride, pink salt (a double salt of stannic chloride and ammonium chlo-

ride) and sodium stannate. Iron salts, with natural extracts containing tannin have been used for a number of years for the weighting of black dyed silks. At the present time the other metals most commonly used are lead, aluminum and chromium (Mathews, 1924).

However, silk weighting is by no means confined to metallic salts. Logwood, gambier, sumac, fustic, quebracho, gall nuts, and pure tannic acid are used to some extent. The organic compounds of tin have been investigated but it was found that a tin salt of formic acid is the only organic tin salt which is suitable for weighting purposes (Scott, 1931).

In spite of the numerous methods of weighting silk 90 per cent of the present day silk weighting is accomplished with the combination of tin, phosphate, and silicate. The process is simple, although the chemical reactions are very complex and are not completely understood. The silk is put in a solution of stannic chloride made acid with hydrochloric acid. Some of the stannic chloride is absorbed by the silk and is fixed on the fiber by washing in clear water so the soluble stannic chloride is hydrolyzed to insoluble stannic acid. The silk is then transferred to an alkaline bath containing disodium phosphate and a complex tin-phosphate compound is formed. The treatments with stannic chloride and sodium phosphate may be repeated a number of times. Finally the silk is immersed in a solution of silicate which increases the weight by the formation of a complex tin-sili-

co-phosphate (Scott, 1931).

Silk put in a stannic chloride solution will acquire an increase in weight. It is assumed that silk has the property of forming an additive compound with stannic chloride and that this compound is subsequently hydrolyzed, releasing the chlorine as free hydrochloric acid. There is a definite limit to the amount of stannic chloride which a given amount of silk will take up from one bath; however this same silk can take up a second charge of stannic chloride by an intermediate washing and consequent hydrolysis of the first charge. The silk fiber does not play any part in these subsequent treatments with sodium phosphate and sodium silicate. Stannic acid is changed into stannic hydroxide in an alkaline solution which reacts with sodium phosphate and sodium silicate. The exact composition of the tin silico-phosphate has never been definitely determined (Scott, 1931).

The results of recent work show that even pure silk is affected by both ultra-violet radiation and perspiration. This is probably due to its chemical and physical structure. Raw silk is made up of two transparent filaments of fibroin, cemented together by sericin. Degummed silk is pure fibroin, a protein made up of the four elements, carbon, hydrogen, nitrogen and oxygen, the exact composition of which is not known (Mathews, 1924). Recently Morley and Sisely (1929) reported traces of sulphur in fibroin.

APPARATUS USED

A Standard Luxor Model Alpine Sun Lamp was used as a source of ultra-violet radiation. The lamp had a quartz mercury arc and was adjustable to various heights.

A heat cradle of the type used for pathological cases in a hospital was used for collecting perspiration. The pH of the perspiration was taken by means of the quinhydrone electrode.

A Lowinson's micrometer was used for counting the threads; the thickness was determined with a Randall and Stickney thickness gauge. Materials to be dried to a constant weight were placed in weighing bottles and heated in an Emerson conditioning oven; all weighings were made on a chainomatic balance.

The breaking strength determinations were made with a Combination Scott Tester, the jaws of which were set three inches apart. The Scott Tester was installed in a room equipped with a Carrier Unit Air Conditioner. A relative humidity of 64 to 66 per cent at a temperature of 69°F. to 71°F. was maintained.

SELECTION OF MATERIALS AND PREPARATION OF SPECIMENS

The materials used for testing were prepared in the research laboratories of Cheney Brothers under known conditions.

A plain woven silk crepe was selected for this work. A

portion of the degummed fabric was treated with a tin phosphate silicate weighting. It contained 35.98 per cent of weighting. Pieces of the unweighted and weighted silks were dyed black. Specimens used in this study were prepared from the two undyed and the two dyed fabrics according to the standard specifications for the strip method as given by the American Society for Testing Materials (1930).

Each specimen was cut six inches long and one and one-eighth inches wide and raveled to one inch in width. In order that all specimens would be fairly representative of the fabric they were not cut nearer to the selvage than one-tenth the width of the material (Bureau of Standards, 1929).

METHOD OF PROCEDURE

Treatment of Specimens

Five warp and five filling strips of each material were used for a test. All specimens were kept between folded sheets of filter paper in the darkened conditioning room except for the periods of exposure to ultra-violet radiation.

The specimens were divided into sets to designate the conditions under which the tests were to be made. Each set was subdivided according to the periods the specimens were to be treated.

The periods and conditions under which the specimens were treated varied as follows:

Set I. Specimens untreated--controls

- Set II. Specimens saturated with perspiration
- A. Saturated once
 - B. Saturated twice, with a 48 hour interval between saturations
 - C. Saturated three times, with 48 hour intervals between saturations
- Set III. Specimens exposed to ultra-violet radiation
- A. Exposed for one 30 minute period
 - B. Exposed for two 30 minute periods with a 48 hour interval between exposures
 - C. Exposed for three 30 minute periods with 48 hour intervals between exposures
 - D. Exposed for one $1\frac{1}{2}$ hour period
- Set IV. Specimens saturated with distilled water and exposed to ultra-violet radiation while moist
- A. Exposed for one 30 minute period
 - B. Exposed for two 30 minute periods with a 48 hour interval between exposures
 - C. Exposed for three 30 minute periods with 48 hour intervals between exposures
 - D. Exposed for one $1\frac{1}{2}$ hour period
- Set V. Specimens saturated with perspiration and exposed to ultra-violet radiation while moist
- A. Exposed for one 30 minute period
 - B. Exposed for two 30 minute periods with a 48 hour interval between exposures
 - C. Exposed for three 30 minute periods with 48 hour intervals between exposures
 - D. Exposed for one $1\frac{1}{2}$ hour period
- Set VI. Specimens saturated with perspiration and dried before exposure to ultra-violet radiation
- A. Exposed for one 30 minute period
 - B. Exposed for two 30 minute periods with a 48 hour interval between exposures

C. Exposed for three 30 minute periods with 48 hour intervals between exposures

D. Exposed for one $1\frac{1}{2}$ hour period

The breaking strength of set I, which was untreated, was determined and used as a basis of comparison with the treated specimens. Set II, which was used to determine the effect of perspiration on the silks, was treated as follows: A was saturated once with perspiration; B was saturated twice with perspiration, a forty-eight hour interval between saturations; C was saturated three times with perspiration, forty-eight hour intervals between saturations. To determine the effect of ultra-violet radiation on the fabrics set III was exposed to ultra-violet radiation: A for one thirty minute period; B for two thirty minute periods with a forty-eight hour interval between exposures to ultra-violet radiation; C for three thirty minute periods with forty-eight hour intervals between exposures to ultra-violet radiation; and D for a one and one-half hour period. Sets IV, V, and VI were exposed to ultra-violet radiation for the same periods of time that were used in set III. Set IV was saturated with distilled water and exposed to ultra-violet radiation while moist; set V was saturated with perspiration and exposed to ultra-violet radiation while moist; set VI was saturated with perspiration and dried before exposure to ultra-violet radiation.

In order to keep conditions as nearly standard as possible the ultra-violet lamp was turned on at least ten

minutes before samples were exposed. The arc of the lamp was thirty inches above the specimens. Filter paper was used as the background for specimens which were being exposed. The specimens were supported by wooden clips during exposure in order that the ones exposed while moist would not come in contact with the background and have their moisture absorbed. In order to keep the moist specimens moist during exposure curtains, kept saturated with water, were hung around the lamp. The specimens exposed while moist for a long period were re-saturated every half hour.

Specimens to be saturated were placed in sterilized evaporating dishes and covered with the liquid. Specimens saturated with perspiration were dried on a sterilized glass surface. They were conditioned at least two hours before exposure. When more than one exposure or one saturation was made, forty-eight hour intervals elapsed between each treatment. The breaking strength was determined forty-eight hours after the last treatment.

Five specimens were used for each set of conditions. The average of these breaking strengths are recorded in Tables 1 to 4 inclusive.

Physical analyses of the fabrics were made according to the specification given by the American Society for Testing Materials (1930). The results of these analyses are given in table 5.

Table 5. Analyses of Silk Fabrics

Material	: Width :(inches)	: Thickness :(inches)	: Thread count :		: Per cent : weighting
			: (per inch)	: Ends : Picks	
White Unweighted silk)	39 $\frac{1}{4}$.0065	181.0	76.5	00.00
White Weighted silk)	37	.0090	189.1	81.0	35.98
Black Unweighted silk)	39	.0060	179.8	76.4	00.00
Black Weighted silk)	37 $\frac{1}{2}$.0075	186.5	78.7	35.98

Collection of Perspiration

Specimens were saturated with human perspiration which was collected in the following manner. The subject was allowed to remain in a tub of clear water (about 40°C.) from from ten to fifteen minutes, rinsed with warm distilled water and dried with a sterile towel. The subject was placed on a sterilized rubber sheet, and the secretion of perspiration was induced by the use of a heat cradle. The cradle was electrically heated for twenty minutes, the temperature ranging between 40°C. to 45°C., then the patient was allowed

to cool gradually before being removed. The perspiration which had collected on the rubber sheet was poured immediately into a sterilized flask which was packed in ice in order to retard changes which might occur in the perspiration. As soon as possible the pH of the perspiration was determined and the specimens were saturated.

Relative Strength of the Ultra-violet Lamp

There is no satisfactory means of making an absolute determination of the intensity of ultra-violet radiation, but the Hanovia Research Laboratory (Journal of the American Chemical Society, 1925) has worked out a relative determination indicating the change in intensity. The method is based on the fact that pure oxalic acid in aqueous solution, when sensitized by a very small amount of uranyl sulphate, is quantitatively decomposed by ultra-violet rays and ultra-violet only. The amount of decomposition of oxalic acid can be determined by titration with standardized potassium permanganate solution.

A measured portion of oxalic acid-uranyl sulphate solution was placed in a quartz cup, thirty inches from the arc, and exposed to ultra-violet radiation for thirty minutes. The solution was then titrated with N/10 potassium permanganate. After the lamp had been lighted for six hours a portion of oxalic acid-uranyl sulphate solution was again exposed for thirty minutes and titrated against potassium per-

manganate. This procedure was repeated several times in order to test different areas. From the tests made the results indicate the strength of the lamp does not vary over a given period of time, but the radiation in different areas is not of the same intensity.

Analysis of Weighting

The "total weighting" of the silk was taken to be all substances that are not silk fibroin. The procedure given in the American Dyestuff Reporter (1931) as recommended by the United States Bureau of Standards for determining the percentage of weighting in silk material was used. A strip of the fabric to be analyzed was dried to constant weight. This was called weight A. The fabric was then treated with water to remove "finishing materials" and then dried again to constant weight, designated as weight B. The fabric from which "finishing materials" had been removed was treated with hydrofluoric-hydrochloric acid reagent until the metallic weighting was removed. The sample was dried and weighed as before. This was weight C. It represents the weight of silk fibroin and residual ash. The sample was ignited to remove the fibroin and weighed. The weight of the ash was D. The amount of "total weighting" in per cent was determined from the formula:

$$\frac{A-C-D}{A} \times 100$$

DISCUSSION OF RESULTS

Silk is one of the strongest textile fibers, however, due to its chemical and physical structure, it is easily affected by both perspiration and ultra-violet radiation.

White specimens exposed to ultra-violet radiation or saturated with perspiration became slightly yellow. Specimens exposed or saturated more than once seemed to be more discolored after each treatment. Since proteins become yellow when oxidized it may be assumed that an oxidation reaction takes place. Specimens, saturated or exposed several times, were more yellow than the specimens treated for one long period of time without a time interval. It may therefore be assumed that the oxidation continues after treatment. The extent of the oxidation reaction was not studied. Color changes in the black specimens could not be detected because no means was available to study the color change.

The data in tables 1 to 4 inclusive shows that the specimens were affected by the treatments. Perspiration seems to have a greater effect on the breaking strength of the specimens than ultra-violet radiation. Moisture in the presence of ultra-violet radiation has more effect on the breaking strength of the specimens than ultra-violet radiation alone. Specimens saturated with perspiration and exposed to ultra-violet radiation while moist show a greater percentage loss in breaking strength than those which were

allowed to dry before exposure to ultra-violet radiation.

The unweighted silks seemed to be more affected by the treatments than the weighted fabrics. The breaking strength of the weighted fabric was higher than the unweighted. This is probably due to the fact that the weighted fabric was more firm and did not slip when the breaking strength determinations were made.

From the analysis of the perspiration by means of the quinhydrone electrode, the results show that the perspiration used was acid except in two instances. The pH of the perspiration from the same subjects varied from time to time.

The results were not consistent in all cases; this may have been due to several factors. The saturated specimens showed a wide variation in breaking strength, especially the weighted specimens. This was probably due to the varying amount of moisture taken up by the specimens when saturated. The weighted specimens did not take up moisture as rapidly as the unweighted specimens and for this reason may not have been thoroughly saturated. Another factor, probably, was the variation in the position of the specimens under the ultra-violet lamp.

CONCLUSIONS

1. Silk fabrics exposed to ultra-violet radiation are tendered.

2. Silk fabrics saturated with perspiration are tendered.

3. Silk fabrics saturated with distilled water and exposed to ultra-violet radiation while moist are tendered.

4. Silk fabrics, saturated with perspiration and exposed to ultra-violet radiation while moist, are tendered more than when saturated with perspiration and allowed to dry before exposure to ultra-violet radiation.

5. A small amount of weighting does not seem to increase the rapidity with which silk fabrics lose strength when treated.

6. Short exposures to ultra-violet radiation with a time interval between exposures cause greater tendering of the silks than one long exposure.

7. Repeated saturations with perspiration increase the rate of tendering of the silks.

8. Repeated saturations with perspiration followed by short time exposures to ultra-violet radiation cause greater tendering of the silks than one saturation with perspiration and one long exposure to ultra-violet radiation.

From the data obtained no conclusions could be drawn as to the effect of the black dyes used on the unweighted and weighted silk fabrics used.

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