

**THE DEVELOPMENT OF A DURABLE
HEAT-RESISTANT FINISH FOR TEXTILES**

A THESIS

**Presented to
the Faculty of the Graduate Division**

By

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**In Partial Fulfillment
of the Requirements for the Degree
Master of Science in Textiles**

Georgia Institute of Technology

December 1957

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HEAT-RESISTANT FINISH FOR TEXTILES

APPROVED:

Date Approved by Chairman: Oct 14, 1927

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ACKNOWLEDGMENTS

A sincere expression of gratitude is offered to my wife, without whose understanding and assistance this study could not have been made; to Dr. James L. Taylor of the Georgia Institute of Technology Textile School, who actively directed and guided the work performed in this study; to Mr. J. W. McCarty and Dr. William Postman for their valuable suggestions and advice; and to Mr. Jack Skelton and Southern Mills Inc. of Atlanta, Georgia, who provided some of the materials used in the laboratory research phase of this investigation.

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THE DEVELOPMENT OF A DURABLE
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SUMMARY

It has been found that laundry press covers of the best quality may be expected to last about six weeks in a laundry operating at normal capacity. This short life of the covers is a result of many factors working together with a synergistic effect. Some of the factors which cause the rapid deterioration of the fabrics are abrasion, high temperatures, water and steam, flexure, stretching, and residual laundry chemicals. Probably the factor which is the most responsible for the covers' short life would be the effect of the high temperatures to which they are subjected.

The purpose of this investigation was to develop a new finish for nylon, Dacron, or cotton which would increase the heat-resisting properties of these textiles to a greater degree than at present. It was found that nylon cloth which had been treated with a phenolic resin compound was among the most durable cover materials now available. For this reason, it was decided that this study would include only phenolic compounds which have structures similar to the structure of pure phenol.

Studies were made on the influence of three variables:

concentration, time, and temperature. The chemicals used were: pyrogallol, phloroglucinol, catechol, para-nitrophenol, and beta-naphthol. Boric acid and para-quinone are patented treatments for a heat-resistant finish which were reproduced in this research for comparative purposes. A sample of Southern Mills Pyron H fabric and a sample of Kohnstamm fabric was also used for comparative purposes. These two fabrics are commercial products of a good quality which have been treated with a heat-resisting finish and used for laundry press covers.

Concentrations of 0.5 per cent and 1.0 per cent were used with time variations of 30 minutes, 60 minutes, and 120 minutes. The temperatures used for treating the fabrics were 65°C. and 90°C.

In order to develop a commercially feasible process, various methods of application were also used. Some fabrics were treated in a bath solution and others were treated with the same concentration of solution by a padding operation. A small laboratory padder was used for this operation.

The method used to heat the treated samples was a specially designed hot-plate tester. After the samples were heated a predetermined length of time, they were then tested for tensile-strength on the Scott Model Q Horizontal tester. The cut strip method was used for the tensile-strength procedure.

Multiple hot-plate tensile-strength determinations

indicated that tests involving 20 minutes hot-plate contact at 230°C. and 240°C. gave good reproducibility of results and showed definite distinctions in the tensile-strength values between various fabrics. Therefore, these two testing temperatures were used throughout the work.

From the data obtained, it was concluded that pyrogallol and phloroglucinol produce results of approximately equal magnitude on nylon fabrics. From a standpoint of the initial cost of the treatment, pyrogallol would be recommended. Concentrations of pyrogallol which gave good results vary from 0.5 per cent to 1.0 per cent (by weight). Both the bath treatments and the padding procedures produced a heat-resistant finish on the nylon fabrics which was better than the two commercial fabrics tested. These results were comparative figures from the hot-plate test only. The service performance characteristics remain to be determined.

CHAPTER I

INTRODUCTION

Historical.--Today, laundry press covers of the best quality may be expected to last about six weeks in a laundry operating at normal capacity. This is a result of the severe service conditions which the covers must withstand. The fabric of these covers is deteriorated very rapidly due to the combined effects of abrasion, stretching, flexure, water and steam, high temperatures, and residual laundry chemicals. Nylon fabrics which are finished with phenolic resin compositions are among the most durable cover materials presently available (1).

The earliest recorded process for a heat-resistant finish found in the patent literature was filed December 14, 1911, by Strickler (2). The treatment consisted of a textile fabric as a base, a rubber solution, and finely ground particles of asbestos and mica incorporated therein. The asbestos and mica particles provided the increased heat-resistance and were held to the fabric by the rubber solution. It is quite apparent that the heat-resistant properties of a fabric produced according to this process are no better than those of the rubber used to bind the asbestos to the fabric. When the rubber becomes hot enough to melt, the increased heat-resisting

properties are lost.

In 1937, Gwaltney, Graybill, and Newton produced a heat-resistant yarn and heat-resistant cord made through the use of their natural gums and waxes (3). The process consisted of twisting the single yarns into a ply, saturating the yarns to soften the waxes and gums of the cotton fiber, and twisting the plied yarns under tension just short of the breaking point to fuse the fibers with the softened gums and waxes retained therein. This process would be useless with the modern finishing operations which the plants employ. The scouring and washing operations of the normal finishing procedures would remove the waxes and gums and also any heat-resisting features provided by them.

The above treatments were for cotton fabrics and yarns only. It was not until June, 1939, that the first treatment appeared for a heat-resistant finish on a fabric other than cotton. This treatment was filed by the Canadian Industries Limited with the Canadian Patent Office (4). The treatment consisted of stabilizing regenerated cellulose yarns against deterioration by heat through the process of impregnating the yarns with urea to at least three per cent by weight of the yarns; air drying the impregnated yarns; and baking the yarns at a temperature above the melting point of the urea under such conditions that volatile products are allowed to escape from the yarns. Another patent was filed at the same time by the Canadian Industries Limited for a treatment to stabilize

cotton or cellulose yarns and fabrics against deterioration by heat through the process of impregnating the yarns with biuret to the extent of at least three and seven tenths per cent by weight of the yarns (5). Biuret is a colorless compound readily formed by heating urea. Therefore, the treatments for both the cotton and the regenerated cellulose above are essentially the same. When the urea impregnated regenerated cellulose is heated, biuret, the ingredient used to impregnate the cellulose yarns, is formed. The end product appears to be the same, even though two approaches are used to reach it.

Only a few years later, in March, 1942, Reese was issued two patents by the United States Patent Office for essentially the same process as outlined above in the Canadian Patents. One of the patents (6) concerned to the stabilization of cellulose yarns against deterioration by heat through the process of impregnating the yarns with biuret to an extent sufficient to stabilize the said yarns against deterioration by heat. The other patent issued to Reese was for a process of impregnating the yarns or fabrics with urea to the extent of at least three per cent by weight of the yarn or fabric (7). The methods of performing the treatments were very similar to the Canadian processes.

Several years elapsed before another patent was applied for and this one was issued in November, 1949. Two patents were issued by the patent office to Dreyfus, Seymour, and Ward who in turn assigned the patents to the Celanese Corporation of

America. One of the patents (8) pertained to the treatment of non-thermaplastic fabrics and the other (9) to cellulose acetate and thermaplastic fibers and fabrics. The treatments used in both patents were identical. The treatment consisted of padding, dipping, or rinsing the textile material in an aqueous solution of boric acid. The aqueous solution contained from 1 to 15 per cent by weight of boric acid. Any solution in excess of the amount desired to be permitted to remain on the textile material could be removed by passing the treated material through squeeze rolls. The textile material with the desired amount of boric acid remaining thereon was dried in any convenient manner, either by passing the fabric through an air chamber of 85° to 110°C. or by passing the fabric over heated drums or cans. After the fabric was dried it was preferable to subject it to hot calendering or decatizing to improve the harsh hand of the fabric and also to eliminate the tendency for an iron to stick to the fabric after the treatment. Temperatures of 110° to 150°C. and pressures of 2000 to 8000 pounds per square inch are usually employed for the hot calendering treatment. The decatizing treatment consisted of winding the fabric on a perforated metal drum with a heavy cotton blanket or apron so that a layer of the cotton blanket or apron alternates with each layer of the fabric. Steam at ten pounds per square inch or more is then passed through the fabric for three to five minutes, whereby the temperature of the fabric is raised to 95°C.

to 110°C. The steam is then turned off and a vacuum is applied to cool the fabric and remove any condensed water present.

Fabrics treated according to the above specifications were supposedly able to undergo ironing at substantially increased temperatures without undergoing scorching or changes detrimentally affecting the fabric. The fabrics also could be dry cleaned, and the improved heat-resisting properties were wholly retained without being impaired in any way. Since boric acid is water soluble, washing would remove it and cause the heat-resistant properties of the fabric to revert back to the original properties before treatment.

The increasing amount of research in the textile field is shown by the number of patents applied for in the latter part of the 1940's and in the 1950's. The rise in the number of patents for heat-resisting finishes for textiles (referred to in this paper) was also great. This indicates that the industry as a whole was busily engaged in expanding research. The previous patent had been issued only a very short time when, in June of 1950, another patent for increasing the heat-resisting properties of a textile fabric was issued. This patent applied for on December 30, 1946 by Gray (10), concerns the treating of a polyamide with a hypophosphorous acid compound. More specifically, the treatment relates to polymers of the nylon type whereby the impairment in strength and the discoloration of nylon by heat can be reduced by incorporating into the nylon a minor amount by weight of a hypophosphorous

acid compound. The hypophosphorous acid compound may consist of the acid itself, which has the formula H_3PO_2 , or a derivative of the acid. These derivatives are the inorganic salts of hypophosphorous acid, aryl or alkyl phosphinic acids, and the salts and esters of these phosphinic acids and of the isomeric phosphonous acids.

In the best method for carrying out the invention, the nylon fabric, yarn, or the like is impregnated with a solution of the hypophosphorous acid compound in a suitable solvent; the excess solution is removed; and the impregnated material is dried. The impregnation can be carried out in conventional fabric treating equipment such as a padder or the fabric may be dipped in solution and then passed through quetch rolls to remove excess solution. The impregnated fabrics may be heat set while they are still wet, or they may be dried before heat setting.

The hypophosphorous compound is incorporated into the nylon in the amount of at least 0.01 per cent and up to 20.0 per cent. The preferred amounts are at least 0.1 per cent and in general it is desired only to use the minimum proportions since the excess tends to decompose during the heat treatment. The impregnation can be carried out at ordinary or elevated temperatures for various lengths of time. The time of immersion should be sufficient to produce uniform impregnation. Usually periods of five to fifteen minutes are suffi-

cient; however, longer times are also operable. The compound may also be blended with the molten nylon, but this method is less satisfactory because hypophosphorous acid and its derivatives tend to decompose at the temperature necessary to melt nylon.

Stokes (11) performed research in 1946 on a process of stabilizing cellulose yarns against deterioration by heat, whereby the yarns were impregnated with 1-phenylbiguanide and then dried. In December, 1951, he was issued a patent on his process which consisted of impregnating the yarn or fabric by immersing it in an aqueous solution of 1-phenylbiguanide of the desired concentration at whatever temperature is required to maintain the 1-phenylbiguanide in solution for a suitable period of time. The yarn may be passed through the solution or the entire skein or fabric may be immersed at once and allowed to remain for the desired length of time. Effective results are obtained by using concentrations of 0.1 per cent to about 1.5 per cent of 1-phenylbiguanide by weight of the yarns. Generally it is preferred to use the agent in amounts from 0.2 per cent to about 0.5 per cent by weight of the yarns.

In 1952 Michaels and Machlis (12) produced a heat-resistant nylon cloth by reaction with quinones. The quinones used were of the class of benzoquinone and anthraquinone, and the treatment was performed by simply immersing the fabric in an aqueous solution of the quinone. The concentration of the solution ranged from 0.5 per cent to 5.0 per cent with a pH

below 7 and immersion in the solution ranged from 12 seconds to 24 hours over a temperature range of 15°C. to 100°C. The most effective treatment was obtained by using a 1.0 per cent solution of benzoquinone in water at room temperature for about 15 hours.

This process would not be feasible for commercial production due to the length of time of the treatment. Therefore, the process may be considerably speeded up by the use of increased temperatures. For example, nylon fabric immersed in a 0.5 per cent solution of benzoquinone in water at 60°C. for 2 hours under agitation produces a very satisfactory product. The fabric should be kept moving at all times to prevent a settling of the benzoquinone in the water. The aqueous solution is prepared by adding steam or boiling water to the powdered chemical to dissolve it. Solvents such as acetone may be used to dissolve the powder and the required amount of water added afterward. It was also found that cloth so treated was impervious to any degenerative effects from starch in the clothing being pressed thereon.

The advances in research were responsible for the production of a heat stabilized cellulose yarn produced by impregnating the yarn with a readily oxidizable metal. Roseveare's patent (13) of September, 1953, was for the treatment of cellulosic and other structures, such as viscose rayon or cuprammonium rayon, with a readily oxidizable metal. It has been discovered that both moisture and oxygen hasten heat degrada-

tion and when they are acting simultaneously a synergistic effect results. This invention, then, is based upon the removal of both water and oxygen by reaction with a metal. The metals preferred for use in this process are those which have only one positive valence and those whose hydroxides are no more water soluble than strontium hydroxide. Such a metal is one which at temperatures of about 150°C. reacts with both air and water to form alkaline products. Certain metals such as zinc, cadmium, tin and lead can form peroxides which tend to degrade cellulose; therefore, they should not be used. Some of the metals which may be used effectively are magnesium, aluminum, strontium, and calcium.

One method of application consists of immersing the textile structure in a proper medium containing the metal suspended in proper concentration. The medium may also contain a finish if desired. In general, any of the well-known finishes and sizes for the textile being treated may be employed in the bath. The immersion may be done by simply dipping the yarn or fabric in the solution and allowing it to remain there for a suitable period of time, or the textile may be impregnated by a continuous passage through the bath. In the continuous process it is important that undue tension of the yarn or fabric is avoided in order to obtain maximum penetration. The speed of the textile through the bath is about 105 yards per minute, so that the period of immersion would be about 0.5 seconds. Other speeds ranging from 100 to 300 yards per minute

may also be used. After passage through the bath the yarn is wound on a reel and allowed to dry. Other drying procedures consist of drying in ovens or with contact driers. Effective results are obtained with concentrations of magnesium from 0.2 per cent by weight on the textile to about 2.0 per cent by weight on the textile. The preferred concentration is from 0.5 per cent to about 1.5 per cent. Similar concentrations of other metals may be used.

Fay developed a treatment whereby a heat-resistant fabric was produced by coating a fabric with a fused composition comprising polytetrafluorethylene and cryolite (14). The process has been limited mainly to glass fabrics because the polytetrafluorethylene coating is usually applied as an aqueous suspensoid which requires heating to at least 327°C. This temperature will burn or char fabrics made from organic fibers; therefore, it is of no value in the particular group of fabrics to which this investigation is directed.

In January, 1956, Roseveare was granted a second patent for his metallic treatment of fabrics to render them heat-resistant (15). The first patent as outlined above was for the treatment of cellulosic structures, whereas the present patent applies only to the treatment of a polyamide structure of the nylon type. The treatment is identical as that outlined above for cellulosic materials. The concentrations, time of immersion, types of metals used, and temperatures used in the treatment are also the same as those listed in a previous

paragraph under his first patent. The principal use of a textile treated according to this method is in the manufacture of tires and reinforced rubber articles. Laundry press covers are listed as a secondary use.

The latest treatment found for producing heat-resistant material involves the property of heat and light reflectance (16). A fabric produced according to the process is used to make fire fighting equipment and would also have little use for the purposes of this investigation. The process uses a textile fabric as a base, with a layer of aluminum powder held to it by use of an adhesive, and then a layer of aluminum sheet held to this by use of a similar adhesive. An adhesive such as polyacrylic acid ester solution or polyvinyl acetate is applied to the textile fabric, the aluminum particles are then added, another layer of the adhesive is added, and finally an aluminum foil having a thickness of about 0.009 mm. is applied.

The preceding treatments are those which have been recorded and in the light of them, the advances due to research are very noticeable. Because of the very keen competition which is found in the textile industry, a large portion of finishing techniques and processes are never recorded and, therefore, they are never known to the competitors or to other members of the industry. This may aid the company which discovers the new process, but it tends to slow down advancement of the industry as a whole. An example of the secretive practices

of the industry is the fact that, although some of the better quality finishes for laundry press covers today are composed of phenol and phenolic resin compounds (1), no patents were found involving these finishes and also nothing was found in the literature referring to such a finish.

Purpose of this investigation.--The purpose of this study was to develop a new finish for nylon, Dacron, or cotton which would increase the heat-resistant properties of these textiles to a greater degree than available with the present known heat-resistant treatments. Secondary objectives were to develop a cheaper finish than presently available which would provide the same service conditions and possibly to develop a finish which would be both cheaper and provide better service conditions than those now known. Since phenolic resin compounds are among the better treatments used in the production of laundry press covers today, it was decided to investigate the effect which compounds of a similar molecular structure would have on increasing heat-resistance of the above named fabrics. Several chemicals with a molecular structure similar to that of phenol were applied to the fabrics in various concentrations, times, and temperatures and the results investigated after the treatment was completed.

After the fabrics had been treated, they were then allowed to condition and scorched through the use of a tester devised mainly for that purpose. After scorching, the samples were again conditioned and tested to determine the per cent

loss in strength. Preliminary screening tests were performed on the fabrics to determine which of the chemicals gave the best results. The concentrations, temperature, and time ranges were then set to cover a broad scope on the chemicals which gave the better results.

From these studies it was felt that a better finish could be developed by mixing some of the known chemicals and through the use of the vast improvements in the resins and chemicals now available to the finisher.

CHAPTER II

THEORETICAL CONSIDERATIONS

Cotton.--The cotton fiber is a long single cell closed at one end. It has the appearance of a narrow flat ribbon with spiral twists and is the purest form of natural cellulose. Cotton is affected by heat at about 150°C ., and if the heat is maintained for a long time the effect is very noticeable. The cotton begins to scorch even in a short time at about 170°C . If cotton is distilled out of contact with air, it chars and decomposes to yield small amounts of gaseous products, acetic acid, and alcohol. Cotton burns freely at high temperatures in air leaving no bead (17).

The temperature of disintegration or decomposition of the cotton fiber is an important one in connection with various manufacturing operations in the preparation of cotton fabrics such as singeing, calendering, hot pressing, and other finishing operations where heat is employed. Heat is also used in drying the cotton after almost every operation or process where the fabric has been treated with solutions such as in scouring, rinsing, dyeing, bleaching, printing, and soaping. Cotton, being a vegetable fiber, is much more resistant to the general effect of heat than is the case with the animal fibers such as silk and wool.

So resistant to heat is cotton, that it is not until

the temperature has risen as high as about 160°C. that a structural disintegration of the fiber takes place. Matthews stated in a paper written in 1923 that some difference is encountered in the destructive action of heat on cellulose depending upon whether dry or moist heat is used (18). He claimed that the destructive action was greater if moisture was not present. This statement appears to be contrary to the modern theories surrounding the effects of heat and moisture on cellulose. Roseveare states that it has been discovered that both moisture and oxygen hasten heat degradation and that when they act simultaneously a synergistic effect results (13). Little also points out that one of the factors which influences the rate of tendering by heat is the initial moisture content of the cellulose and of the surrounding atmosphere (19). He indicates that the breaking strength appears to be a function of the relative humidity at which the samples are exposed. The damage is greater at high humidity. Thus, it can be pointed out that the views regarding the effects of heat and moisture have not only changed in the past thirty years but have been completely reversed. The more advanced research methods and equipment available to the research laboratories today are probably the leading factors in determining the change in the two theories.

It should also be pointed out that cellulosic substances are readily degraded by acids, oxidizing agents, enzymes, heat, and light. With enzymes, heat, and light the processes of

breakdown are complicated and the chemical mechanisms of the reactions are not completely understood. In order to develop a chemical treatment to prevent deterioration by heat of the cellulose, it is necessary that the chemicals employed are not of the classes mentioned in this paragraph, because a deterioration of the fabric would occur merely by their application. In view of the well-known sensitivity of cellulose to acids, it is safe to say that any treatment for cotton textiles should not leave an acidic residue on the fabric. Also, oxidation generally proceeds most rapidly near a pH of 7 and for this reason chemicals which will oxidize cellulose are never applied in a neutral bath (20).

Some distinction must be made between the effects of heat on cotton in the raw state and cotton in the bleached state. In the raw state, cotton contains waxes, gums, and other impurities which, when heated, undergo some degree of decomposition and a resulting discoloration. Also, the decomposition products of these impurities may engender an acid condition in the fiber which leads to a weakening and gradual breakdown. Bleached goods, on the other hand, bring in other factors which may result in a serious weakening of the fabric when subjected to ordinary temperatures for a long time. If cotton has been overbleached and there is a formation of oxycellulose, the effect of even moderate heat will be quite marked (18).

The difficulties involved in establishing the course of

the chemical degradation of cellulose by heat are emphasized by the lack of specific information on the subject. Work has largely been limited to discussion of the properties of cellulose degraded by this means. Some of the properties of cellulose degraded by heat are a loss in weight, elasticity, and strength. The fabric also becomes brittle and harsh to the feel with a discoloration of the normal white of the fabric. The losses indicated above are directly dependent upon the temperature applied and the length of exposure time. Cotton begins to yellow at 120°C. after five hours and begins to decompose at 150°C. with a loss in strength above 149°C. (21).

Nylon.--Nylon was developed in the laboratories of the E. I. du Pont de Nemours Company in Wilmington, Delaware, by a group of research organic chemists under the direction of Dr. Wallace H. Carothers. It is a protein-like substance, that is, a nitrogenous substance composed of long chains, a high polymer. The polyamides discovered by Carothers are obtained by condensing diamines and dicarboxylic acids. The nylon fiber of commerce is obtained by condensing hexamethylene diamine and adipic acid. A product made in this manner would normally contain carboxyl and amino groups in equal numbers at the end of the polyamide chains. As manufactured, however, this is not the case. A small amount of monocarboxylic acid is added to stop the polymerization and terminate the growing chains, which it does by reacting with the amino groups. All the amino groups are not blocked and the finished product contains free

carboxyl groups together with a smaller number of free amino groups and some terminal amide groups (22). These groups should be carefully noted because they probably account for the ability of nylon to react with a chemical.

It is also necessary to use a chemical which will not seriously degrade the fabric being treated. Nylon has extremely good resistance to most chemical reagents, especially organic compounds; therefore, the chances of selecting a chemical which will not degrade the fabric is very good. Nylon is inert to organic reagents such as aldehydes, ketones, alcohols, soaps, and many organic acids. Ordinary organic solvents such as gasoline, benzene, chloroform, carbon tetrachloride, and trichloroethylene have no effect on nylon. Among the solvents for nylon, some of the best are concentrated formic acid and the phenols: phenol (carbolic acid), metacresol, xylenol, and cresylic acid. Nylon is somewhat more reactive with certain inorganic reagents. Caustic alkalis have no effect on it but it loses strength when treated with ordinary oxidizing agents or mineral acids.

The heat-resistance of nylon is satisfactory, especially in the absence of oxygen. A temperature of 200°C. has no effect on nylon yarn in an oxygen-free atmosphere after three hours, but at 225°C. after three hours some loss in strength is found. There is a considerable loss in strength at 245°C. (23). Another source gave 482°F. as the melting point of nylon which is approximately 250°C. The same source pointed

out that nylon begins to stick at 235°C. No data were given pertaining to losses in strength due to the effect of heat on the fabric (21).

Achhammer performed an investigation on nylon to determine which bonds are broken when nylon molecules are ruptured due to the effect of high temperatures (25). Nylon samples of 66/6 and 610/6 polymers were heated at 420°C. in a high vacuum for twenty minutes and the pyrolysis products--gases, liquids, and non-volatile residues--were collected separately. Only the gaseous products, which amounted to approximately 5.0 per cent of the total were analyzed. With the exception of carbon dioxide and carbon monoxide, the volatile products consisted of aliphatic and aromatic hydrocarbons, saturated and unsaturated, of less than 7 carbon atoms to the molecule and cyclopentanone. Carbon dioxide was obtained in ten times the amount that could be accounted for as originating from the end-groups of the linear nylon polymer chain. Assuming the validity of the conventional acid amide formula for nylon, $(-CO-NH-(CH_2)_6-NH-CO-(CH_2)_4-CO-NH-)_n$, and on the basis of the information uncovered by them, it was concluded by Achhammer that the polymer chain breaks at the -C-N- bond of the peptide group and the nitrogen atom remains attached to the polymer molecule.

More work was performed in 1952, a year after Achhammer had performed his experiments, by using Oxford nylon cloth "41" and heating it from 280°C. to 400°C. A Linberg oven was used

to heat the samples and the pyrolysis time varied from 1 hour to 18 hours. The charred nylon samples from above were extracted in a Soxhlet apparatus with ethanol. The per cent of alcohol soluble material increased as the temperature increased. On analyzing the ethanol soluble and insoluble portions of the residue it was found that the results were nearly similar, which seemed to suggest that nylon depolymerizes on heating. It was also discovered that considerable amounts of ammonia was formed in the pyrolysis of nylon which was incompatible with the assumption of Achhammer that the main reaction mechanism is the breaking of the $-C-N-$ bond. The work discussed at this time shows that the breaking also occurs at the $-CH_2-CO-$ bond, and that this reaction should be considered as one of the possible reactions of nylon pyrolysis. The carbon dioxide and ammonia formation in nylon pyrolysis cannot be explained conveniently from the accepted acid amide formula for the nylon polymer. The nylon fabric used in this work contained more oxygen but less carbon and nitrogen than calculated for the nylon repeating unit $-C_{12}H_{22}O_2N_2-$. The data supports the observation that the nitrogen and oxygen are removed in the beginning and hydrogen at the end of the reaction. The hydrogen is apparently given off in a combined form as low weight aliphatic Hydrocarbons, since butene was identified (24).

These two papers appear to be the only ones published on the effect of pyrolysis on nylon. Since there is a dis-

agreement between the two works, it is then up to the individual concerned to draw his own conclusions as to which paper he considers as being more representative of the facts.

Dacron.--Dacron is the trade-mark for a polyester fiber manufactured by the Du Pont Company from a polymer called polyethylene terephthalate. The early research work by Dr. Carothers in the United States that led to the development of nylon was responsible for the outgrowth of the fiber which was developed in England under the trade-name of "Terylene". Dacron has excellent resistance to oxidizing agents and is not degraded by bleaching treatments normally used for other fibers.

Dacron has a remarkable resistance to heat. There is very little discoloration or degradation due to high temperatures. An example of the good heat-resisting properties of the fiber is evidenced by the fact that only 74 per cent of the original strength is lost after thirty days in a hot oven at 149°C . The sticking temperature of the Dacron is 238°C . and the melting temperature is 249°C . Dacron melts as long as a flame is applied directly to the fabric. However, the fabric ceases to melt and there is no tendency for further flame propagation after the flame is removed (25). Another source gives the melting point of Dacron at 250°C . and a sticking temperature of 235°C . (21).

Due to the fact that Dacron is a relatively new fiber and has been available to the public for only a few years,

there is very little information in the literature pertaining to the properties of the fiber. This is especially true of the heat-resistant properties and of the effects of heat on the fiber. No information was available on what happens to the fiber when heat is applied nor where the polymer chain breaks as a result of higher temperatures.

Chemicals Used in this Research

The properties of the various chemicals used in this thesis are given below under separate headings for each of the chemicals. It was felt that a knowledge of these properties would help give a better understanding of the reactions which take place between them and the fibers. Figure 1 shows the molecular structure of each of the chemicals used.

Phenol.--Phenol was used in the form of a phenol-formaldehyde resin in this research and not in its pure form. An analysis of some of the better known laundry press cover fabrics revealed that there was substantial free phenol plus phenol-formaldehyde resin present. Therefore, it was decided to try other molecules with a structure similar to that of phenol. This is why phenol is discussed in this chapter and its structure given in Figure 1.

Phenol is a hydroxyl derivative of an aromatic hydrocarbon in which the -OH group is attached to a nuclear carbon atom. Phenol crystallizes in colorless prisms and has the characteristic odor of carboic acid, which is another name

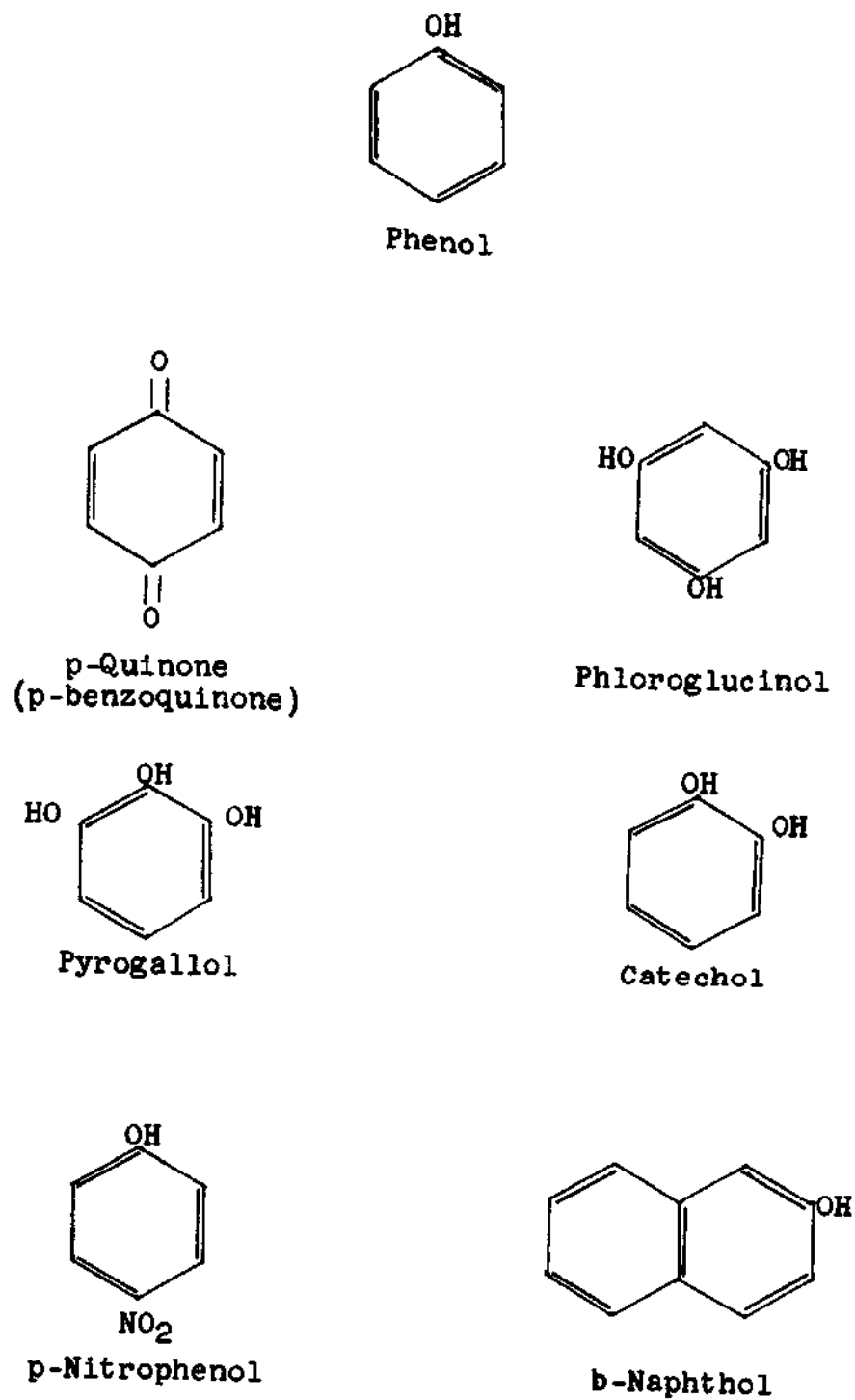


Figure 1. Structural Formulas of Chemicals used in this Study.

for it. Phenol is definitely an acid, but a very weak one. Crystals of phenol and its concentrated solutions will produce painful burns on contact with the skin and even dilute solutions will act as a poison when taken internally. The largest single use of phenol is in the manufacture of synthetic resins of the Bakelite type. The -OH group of phenol makes the nucleus easily oxidized; therefore, it will react as a reducing agent. Vickerstaff states that phenol has an affinity for Dacron, being absorbed by hydrogen bonding (26). Van der Waals forces must also be considered as a possible bonding force.

Para-quinone.--Para-quinone, or para-benzoquinone as it is commonly called, is a patented treatment for increasing the heat-resistance of textiles. Several methods of application were used in this work to produce a fabric which could be used to compare the effectiveness of the treatment it was hoped would be developed.

Para-quinone crystallizes in deep yellow needles which dissolve readily in ether and in alcohol but dissolve only sparingly even in hot water. It contains two carbonyl groups, and enters into various reactions characteristic of the ethylenic double bond.

Phloroglucinol.--Phloroglucinol, a 1,3,5-trihydroxybenzene, is of no commercial importance. It is a colorless crystalline solid which is usually regarded as a trihydric phenol and has a melting point of 219°C. It is very soluble in alcohol and

ether but only sparingly soluble in water. At 25°C. only 1.13 grams are soluble in 100 ml. of water. Its absorption spectrum resembles that of other phenols but on the other hand, it forms a trioxime which shows an evidence of three carbonyl groups. No one has been able to isolate isomeric forms of phloroglucinol to correspond to the two possible formulas. Phloroglucinol may be classed as a reducing agent but it is not as good for this purpose as some of the other chemicals used herein.

Pyrogallol.--Gallic acid yields pyrogallol on heating. It is used as an intermediate for a number of azo dyes and is also a powerful reducing agent for absorbing oxygen. It is a 1,2,3-trihydroxybenzene with a melting point of 134°C. It is readily soluble in water, 62.5 grams dissolving in 100 ml. of water at 25°C.

Catechol.--Catechol is a 1,2-dihydroxybenzene with a melting point of 105°C. The compound is a solid and its water solubility is greater than that of a monohydric phenol. Approximately 45.1 grams will dissolve in 100 ml. of water at 20°C. It is a good reducing agent and its reducing action makes it useful as a photographic developer.

Para-nitrophenol.--The ortho and para-nitrophenols may be prepared by the nitration of phenol with dilute nitric acid. The product formed consists of approximately equal parts of ortho-nitrophenol and para-nitrophenol. Separation is affected by steam distillation, the ortho-nitrophenol alone being

volatile. Para-nitrophenol crystallizes in colorless needles with a melting point of 114°C . The solubility in water is fair, 1.6 grams dissolving in 100 ml. of water at 25°C . At 90°C ., 26.9 grams dissolve in 100 ml. of water. The ortho-nitrophenol forms canary-yellow crystals. The presence of the nitro group greatly increases the acidity of the compound and the nitrophenols are more acidic than phenol and most of its simple derivatives.

Beta-naphthol.--The hydroxy derivatives of naphthalene may be prepared from naphthlene by a process identical with that used for obtaining phenol from benzene. Beta-naphthol is a colorless solid with a melting point of 122°C . and a boiling point of 285°C . It resembles phenol in its general reactions; it is a weak acid, soluble in sodium hydroxide solution but not in sodium bicarbonate solution. It is prepared mainly for use in the manufacture of azo dyes. Beta-naphthol is freely soluble in ether and most other organic solvents. Solubility in water is very limited but is somewhat increased by alkalies. Approximately 1.25 grams will dissolve in 100 ml. of hot water.

Boric Acid.--Boric acid is a weak acid, since, in water solution, it is only slightly dissociated into its ions. The solid exists in white, slippery, crystalline scales. When boric acid is heated, it loses water, yielding tetraboric acid, $\text{H}_2\text{B}_4\text{O}_7$, and finally boric anhydride, B_2O_3 . It decomposes at 185°C . and is moderately soluble in hot water; 40.2 grams will

dissolve in 100 ml. of water at 100°C. The structure of boric acid is not given in Figure 1 because it is an inorganic compound with no ring structure or similarity to the other compounds presented.

CHAPTER III

MATERIALS AND EQUIPMENT

The fabrics used were spun nylon, Dacron, and cotton of the following specifications:

	<u>Cotton</u>	<u>Nylon</u>	<u>Dacron</u>
Weight, Lbs./Lin. Yd.	.52	.42	.56
Breaking Strength, pounds, (out strip test, 1 x 6 inch sample)			
Warp	114.60	159.30	185.80
Weave	sateen	plain	plain
Construction			
Ends/inch	98	37	35
Picks/inch	64	39	37
Width, inches	53.0	57.0	65.0
Count			
Warp	14's	16/2	12/2
Filling	14's	16/2	12/2

With the exception of the hot-plate tester all instruments and machinery used were standard testing equipment in the laboratories of the A. French Textile School. The hot-plate tester was devised by the Georgia Tech Experiment Station for the purpose of evaluating the comparative properties of heat-resisting finishes on fabrics. A piece of 5/8 inch steel

plate was cut to the dimensions of $5\frac{1}{2}$ by $8\frac{1}{2}$ inches. A small hole was drilled into one edge of the plate $1\frac{1}{2}$ inches deep for use as a thermometer well. The plate was placed on a Linberg H-1 hot-plate and a padded board of the same dimensions as the steel plate was loaded on top and weighted with a thick bar of steel to an aggregate weight of six pounds. This weighted board was used to press the specimens against the upward face of the steel plate on the hot-plate. A standard laboratory thermometer with a maximum range of 360°C . was used to check the temperature of the steel plate.

All tensile strength tests both before and after hot-plate exposure were made on a Scott Model Q Horizontal Tester equipped with 2 inch jaws.

Stainless steel beakers of a three liter capacity were used to treat all fabrics by the bath process. The proper formulas were also mixed in and the fabrics passed through the solutions in these beakers for the padding operations. A laboratory padder was used to pad the fabrics which were treated in that manner.

CHAPTER IV

PROCEDURE

Sample preparation.--Strips $6\frac{1}{2}$ inches wide were cut filling-wise from each of the three fabrics; each strip was then cut in half to yield pieces approximately 30 inches long. The $6\frac{1}{2}$ by 30 inch samples were treated and then cut into 9 inch lengths for subsequent testing. One nine inch length was heated on the hot-plate tester at 230°C . for 20 minutes and one was heated at 240°C . for 20 minutes. After allowing each sample to condition for at least 24 hours after heating, the heated samples were cut into 1 inch strips for tensile strength tests. The tensile strength testing procedure followed specifications set forth in the ASTM Standards for Textile Materials. In order that treated samples might be compared with the heated samples, and the strength loss of the heated samples determined, the third nine inch length of fabric which was not heated was cut into one inch strips and tested for tensile strength. This same procedure was followed for testing the untreated fabrics to use as a comparison for the effectiveness of the chemicals employed to increase the heat-resistance.

Testing procedure in general.--All chemicals employed in this research were applied to the fabrics from an aqueous substance. For purposes of economy and safety, the solvent in all cases

was hot water. A portion of the chemicals tested have a limited solubility in water. The concentrations of chemicals in the solutions were so small that no difficulty was encountered in dissolving the chemicals in hot water.

The various treatments performed are listed according to treatment numbers in the tables found in Chapter V. For this reason, each treatment number will be dealt with separately in this chapter; each concentration, time, and temperature range are given for each number.

Treatment 1d consisted of dissolving 0.5 per cent (by weight) of para-quinone in hot water. The fabrics were immersed for 2 hours at 60°C., washed thoroughly to remove excess solution, dried, conditioned, and tested.

Treatment 1e, according to the patent, was supposedly the best treatment for nylon with para-quinone. One per cent (by weight) of para-quinone was dissolved in hot water. The fabrics were immersed for 15 hours at room temperature, washed thoroughly to remove excess solution, dried at room temperature, conditioned and tested.

Catechol was used for treatment 2. One per cent (by weight) of catechol was dissolved in water. The fabrics were immersed for 2 hours at 65°C., washed, dried at room temperature, conditioned and tested.

Treatment 3 was also a patented treatment. It consisted of dissolving 5.0 per cent (by weight) of boric acid in water, immersing the fabrics in the solution for a few minutes,

and then removing excess solution by running the goods through a padder. The fabrics were then dried in an oven between 85°C. and 110°C., conditioned, and tested.

Treatments 4p and 4w both consisted of applying para-nitrophenol to the fabrics. One per cent (by weight) of para-nitrophenol was dissolved in water and the fabrics were immersed in this solution for one hour at 65°C. At the end of the hour, sample 4p was run through the squeeze rolls of the padder to remove excess solution and sample 4w was washed to remove the excess. Both samples were then dried, conditioned, and tested.

Treatments 5p and 5w were analogous to 4p and 4w. Number 5p was padded to remove excess solution and 5w was washed to remove the excess. The treatments consisted of immersing the fabrics in a 1.0 per cent (by weight) solution of pyrogallol for one hour at 65°C. After padding and washing, the samples were air dried, conditioned, and tested.

A 1.0 per cent (by weight) solution of phloroglucinol in water was used for treatment 6. The fabrics were immersed in the solution for 1 hour at 65°C., washed to remove excess solution, air dried, conditioned, and tested.

Treatment number 7 also consisted of using phloroglucinol in water. A 0.5 per cent (by weight) solution was used and the fabrics were immersed for only 30 minutes at 90°C. A 0.5 per cent (by weight) of Triton X-100, a concentrated wetting agent manufactured by the Rohn and Haas Company, Philadelphia,

Pennsylvania, was also added to the bath. At the end of the treatment, the fabrics were washed, air dried, conditioned, and tested.

Treatment 8 is identical with treatment 7 with the exception that pyrogallol is used instead of phloroglucinol.

Treatment 9 is identical with treatment 8 except no wetting agent is used in the bath. Pyrogallol is the chemical used here also.

Treatment 10 was performed by mixing 1.0 per cent (by weight) of pyrogallol in water with 0.1 per cent (by weight) of Triton X-100; immersing the fabrics for one hour at 65°C.; washing; air drying; conditioning; and testing.

Treatment 11 contained phloroglucinol and was identical with treatment 7 except no wetting agent was used.

Phloroglucinol was the base for treatment 12 also. A 0.5 per cent (by weight) of phloroglucinol was dissolved in water along with a 0.1 per cent (by weight) of Triton X-100. The fabrics were immersed for one hour at 90°C., washed, air dried, conditioned, and tested. Treatment 13 was a repetition of number 12 with no wetting agent in the bath.

Treatment 14 consisted of padding a specially prepared phenolic resin solution to the fabrics, curing the fabrics in an oven at approximately 177°C. for ten minutes, washing, conditioning, and testing. The laboratory preparation of the resin followed the recommended procedure of D'Alelio (27). The formulation consisted of 250 grams of phenol, 150 grams of

37 per cent formalin, 4 grams of NaOH, and 20 grams of water. The resin obtained from this mixture was then mixed into the following formula and padded to the fabrics. The resin comprised 20 per cent (by weight) of the mix, 0.1 per cent was Triton X-100, 10.0 per cent carbowax 4000, and 69.9 per cent water.

Technical grade beta-naphthol was used for treatment number 15. One per cent (by weight) was dissolved in water and the fabric was immersed in the solution for 30 minutes at 90°C., washed, dried, conditioned, and tested.

Treatments 16, 17, 18, and 19 are the same as treatments 5w, 8, 9, and 10 with the exception that they are allowed to dry after the treatment before washing. All concentrations and other operations remain the same.

Treatments 20a, 20b, and 20c were all performed using the same solution. One per cent (by weight) of para-quinone was dissolved in water and samples of nylon were dipped in the bath and then run through the squeeze rolls of the laboratory padder. Number 20a was dipped in the solution once, 20b was dipped twice, and 20c was dipped three times. The samples were dried and then washed after which they were again dried, conditioned, and tested. A sample of Dacron was run according to 20c only. The temperature of the bath was held at 90°C. while the samples were being dipped in the solution.

A pyrogallol solution was padded on the nylon fabric in treatments 21a, 21b, 21c. One per cent (by weight) of pyro-

gallol was dissolved in water with 0.1 per cent (by weight) of Triton X-100. The bath was held at 90°C. while the samples were being dipped. Treatment 21a was dipped once, 21b was dipped twice, and 21c was dipped three times. After each dip the fabrics were run through the squeeze rolls of the padder to remove excess solution. After padding the samples were allowed to dry and then they were washed, dried, conditioned, and tested. A Dacron sample was run according to 21c.

The 22 series, 22a, b, and c, consisted of dissolving 1.0 per cent (by weight) of para-quinone in water and dipping the nylon fabric one, two, and three times respectively. The fabrics were passed through the squeeze rolls of the padder after each dip and the temperature of the bath was held at 90°C. for all dips. The samples were dried, washed, dried, conditioned, and tested in that order after the padding operation.

The last series of treatment, 23a, b, and c, were also performed on the laboratory padder. One per cent (by weight) of pyrogallol was dissolved in hot water and no Triton X-100 was used in the bath. The temperature of the bath was held at 90°C. for all dips. One dip was made for 23a, two dips for 23b, and three dips for 23c. The fabric was passed between the squeeze rolls of the padder after each dip in the solution. After squeezing out the excess solution, the fabrics were dried, washed, dried, conditioned, and tested in that order.

Testing the treated fabrics.--The fabrics were allowed to

condition for at least 24 hours after they had been dried following the chemical treatments. These fabrics were cut into nine inch lengths at this time and carefully marked in accordance with a predetermined numbering system.

The temperature of the hot-plate tester was checked to insure that the proper temperature was being used. This temperature also was checked periodically during the heating of a sample to insure a constant temperature throughout the duration of the heating. The temperature was held constant within limits of plus or minus 3°C.

One of the nine inch treated samples was placed on the surface of the hot-plate tester at a temperature of 230°C. and allowed to remain there with the weighted board holding it securely against the face of the steel plate for 20 minutes. Another nine inch sample from the same treated fabric was then placed on the hot-plate tester at a temperature of 240°C. and allowed to remain for 20 minutes. A third nine inch sample from the treated fabric was not heated. This procedure was followed for each of the treated fabrics.

The heated samples were allowed to condition for 24 hours before being tested. This testing consisted of cutting the heated samples into five one inch strips according to the ASTM specifications and testing the breaking strength with the Scott Model Q Horizontal Tester.

Prior to making any tests on the treated fabrics, a number of untreated samples were heated at both temperatures and

broken to determine the per cent loss in strength of the untreated goods to use as comparative figures.

All results on the treated fabrics and the untreated fabrics were tabulated, averaged, and the per cent loss in breaking strength calculated. Tables have been compiled showing the comparative results of these strength losses and are included in Chapter V.

A comparison of the treated fabrics with the untreated fabrics and a visual examination was made in each case to determine the changes in the hand and color which resulted from the treatment alone, or from the application of heat to the treated fabrics and the untreated fabrics. These results are also listed in tabular form in Chapter V.

CHAPTER V

RESULTS AND DISCUSSION OF RESULTS

General.--In order to provide data for comparison with results obtained from treated samples, samples of the raw cotton, nylon, and Dacron fabrics were tested for breaking strength. The samples were then heated to 230°C. for 20 minutes and 240°C. for 20 minutes after which the breaking strength for each was determined and the per cent loss in strength calculated. The Southern Mills Pyron H sample and the Kohnstamm sample were both commercially finished nylon fabrics of good quality which are used as heat-resistant fabrics. They were used in this work for comparative purposes only. The standard deviation and coefficient of variation values for each of the untreated samples, both heated and unheated, are found in Table 1. The higher values for the heated samples of nylon appear to be a result of the affect of oxygen on the fabric being heated. It has been mentioned previously that oxygen hastens the degradation of nylon when heat is applied and this theory was evidenced very clearly on the untreated nylon samples. Around the edge of the weight used to hold the sample firmly in place, the fabric was much weaker after heating. Underneath the weight where the oxygen was not available in abundance, the fabric was not weakened to the extent at which the edges were. The

weakened edges of the heated nylon caused the tensile strength of the samples to vary by a considerable amount. This explains the larger standard deviation and coefficient of variation for those particular samples. The oxygen had very little effect on the treated samples which were heated; therefore, the coefficients of variation on all treated samples appeared to fall within suitable limits.

The average tensile strength of the untreated samples before and after heating is presented in Tables 2, 3, and 4. The average tensile strength of the treated samples is also shown in the above listed tables along with the strength loss in per cent due to the effect of the heat on the fabrics.

Table 1. Variation of the Untreated Samples*

Sample	Standard Deviation	Coefficient of Variation
Cotton:		
Unheated	2.30	2.01
230°C. for 20 minutes	5.80	9.44
240°C. for 20 minutes	1.02	2.52
Nylon:		
Unheated**	8.49	5.33
230°C. for 20 minutes**	11.64	14.20
240°C. for 20 minutes--	8.06	12.43
Dacron:		
Unheated	8.03	4.32
230°C. for 20 minutes	5.47	3.41
240°C. for 20 minutes	4.05	4.71

*. Computed from five tests unless otherwise specified.

**.. Computed from twenty tests.

Table 2. Comparative Strength Losses of the Nylon

Treatment	Tensile Force in Pounds*			Strength Loss (%)	
	As Received	No. 1**	No. 2***	No. 1**	No. 2***
Untreated Nylon	159.30 ^a	42.20 ^a	10.20	73.51	93.80
Southern Mills	142.40	126.40	89.00	11.23	37.50
Kohnstamm sample	117.00	69.80	54.60	40.34	53.33
1d. p-Quinone	152.80	140.60	132.60	7.90	13.22
1e. p-Quinone	151.40	149.20	118.50	1.45	19.12
2. Catechol	154.20	119.80	96.20	22.31	37.61
3. Boric acid	154.60	46.80	34.80	69.72	77.48
4p. p-Nitrophenol	157.80	40.60	39.40	74.27	75.03
4w. p-Nitrophenol	126.40	104.40	57.80	17.41	54.27
5p. Pyrogallol	153.80	115.20	64.80	25.09	57.87
5w. Pyrogallol	138.00	128.20	89.40	7.10	35.22
6. Phloroglucinol	158.00	119.20	93.00	24.56	39.87
7. Phloroglucinol	175.40	149.00	142.20	15.05	18.92
8. Pyrogallol	163.40	144.20	132.80	11.75	18.73
9. Pyrogallol	157.60	144.50	108.80	8.31	30.96
10. Pyrogallol	159.20	147.00	126.80	7.66	20.35
11. Phloroglucinol	155.60	148.80	107.00	4.37	31.23
12. Phloroglucinol	165.80	142.00	127.20	14.35	23.28

*. Average of five breaks unless otherwise specified.

**. After hot-plate test at 230°C. for 20 minutes.

***. After hot-plate test at 240°C. for 20 minutes.

..a. Average of twenty breaks.

Note: 1d was treated 2 hours and 1e was treated 15 hours. A p following a treatment number denotes that the sample was padded; w following the number signifies the sample was washed.

Table 2 Continued. Comparative Strength Losses of the Nylon

Treatment	Tensile Force in Pounds*			Strength Loss (%)	
	As Received	No. 1**	No. 2***	No. 1**	No. 2***
13. Phloroglucinol	166.40	151.60	124.80	8.89	25.00
14. Resin #1	174.80	97.20	93.40	44.39	46.57
15. <u>Beta</u> -naphthol	135.00	57.80	29.60	57.18	78.07
16. Pyrogallol	149.60	141.00		5.75	
17. Pyrogallol	148.80	137.60		7.53	
18. Pyrogallol	146.60	136.80		6.68	
19. Pyrogallol	147.00	132.40		9.93	
20a. p-Quinone	151.40	143.40	86.20	5.28	43.06
20b. p-Quinone	153.60	141.50	129.00	7.87	16.02
20c. p-Quinone	153.20	144.20	136.40	5.87	10.97
21a. Pyrogallol	150.00	140.20	121.20	6.53	19.20
21b. Pyrogallol	150.00	131.80	125.80	12.13	16.13
21c. Pyrogallol	151.60	136.60	117.40	9.89	22.56
22a. p-Quinone	147.80	138.20	122.00	6.49	17.46
22b. p-Quinone	148.60	136.00	120.00	8.48	19.25
22c. p-Quinone	147.20	141.20	129.20	4.08	12.23
23a. Pyrogallol	148.20	125.00	107.00	15.65	27.80
23b. Pyrogallol	147.80	127.80	124.40	13.53	15.83
23c. Pyrogallol	139.80	119.40	117.00	14.59	16.31

*. Average of five breaks unless otherwise specified.

** . After hot-plate test at 230°C. for 20 minutes.

*** . After hot-plate test at 240°C. for 20 minutes.

Note: The treatment numbers which have a, b, and c beside them signify that the samples were padded one, two, and three times respectively.

Table 3. Comparative Strength Losses of the Dacron

Treatment	Tensile Force in Pounds*			Strength Loss (%)	
	As Received	No. 1**	No. 2***	No. 1**	No. 2***
Untreated Dacron	185.80	160.60	86.00	13.55	53.71
Southern Mills	142.40	126.40	89.00	11.23	37.50
Kohnstamm Sample	117.00	69.80	54.60	40.34	53.33
1d. p-Quinone	170.80	167.80	127.00	1.75	25.64
1e. p-Quinone	170.20	143.40	130.00	15.74	23.62
2. Catechol	179.40	124.40	69.80	30.66	61.09
3. Boric acid	174.00	146.40	36.20	15.86	79.19
4p. p-Nitrophenol	183.00	158.40	99.60	13.44	45.57
4w. p-Nitrophenol	165.00	163.40	114.00	0.97	30.91
5p. Pyrogallol	179.80	165.80	137.20	7.79	23.64
5w. Pyrogallol	171.20	171.40	144.80	0.12 ^b	15.42
6. Phloroglucinol	175.80	175.80	167.20	0.00	4.89
7. Phloroglucinol	173.80	176.00	161.00	1.27 ^b	7.37
8. Pyrogallol	185.20	177.20	136.40	4.32	26.35
9. Pyrogallol	188.20	175.60	150.60	6.70	19.98
10. Pyrogallol	176.00	175.60	150.60	0.23	14.43

*. Average of five breaks unless otherwise specified.

**. After hot-plate test at 230°C. for 20 minutes.

***. After hot-plate test at 240°C. for 20 minutes.

..b. Denotes a per cent gain in strength.

Note: 1d was treated 2 hours and 1e was treated 15 hours. A p following a treatment number denotes that the sample was padded and not washed; w after a number signifies that the sample was washed.

Table 3 Continued. Comparative Strength Losses of the Dacron

Treatment	Tensile Force in Pounds*			Strength Loss (%)	
	As Received	No. 1**	No. 2***	No. 1**	No. 2***
11. Phloroglucinol	188.00	170.00	158.60	9.57	15.64
12. Phloroglucinol	190.80	189.80	159.40	0.52	16.46
13. Phloroglucinol	186.60	179.60	166.00	3.75	11.04
14. Resin #1	209.60	173.00	142.20	17.46	32.16
15. Beta-naphthol	196.80	156.40	101.60	20.53	48.37
16. Pyrogallol	195.40	180.80		7.47	
17. Pyrogallol	190.00	176.00		7.37	
18. Pyrogallol	184.20	171.20		7.06	
19. Pyrogallol	192.00	169.00		11.98	
20c. p-Quinone	189.00	174.40	153.20	7.72	18.94
21c. Pyrogallol	194.80	173.60	146.00	10.88	25.05

*. Average of five breaks unless otherwise specified.

**. After hot-plate test at 230°C. for 20 minutes.

***. After hot-plate test at 240°C. for 20 minutes.

Note: The c following the treatment numbers signifies that the sample was dipped and padded three times.

Table 4. Comparative Strength Losses of the Cotton

Treatment	Tensile Force in Pounds*			Strength Loss (%)	
	As Received	No. 1**	No. 2***	No. 1**	No. 2***
Untreated Cotton	114.60	61.40	40.40	46.42	64.73
Southern Mills	142.40	126.40	89.00	11.23	37.50
Kohnstamm Sample	117.00	69.80	54.60	40.34	53.33
1d. p-Quinone	94.00	34.80	25.00	62.97	73.40
1e. p-Quinone	109.60	35.80	11.80	67.33	80.11
2. Catechol	94.80	38.60	28.60	59.28	69.83
3. Boric acid	102.40	41.00	29.40	59.96	71.28
4p. p-Nitrophenol	104.40	34.40	26.80	67.05	74.33
4w. p-Nitrophenol	90.40	57.00	37.60	36.95	58.41
5p. Pyrogallol	100.00	45.80	32.20	54.20	67.80
5w. Pyrogallol	101.00	48.20	38.20	52.28	62.18
6. Phloroglucinol	101.80	35.60	21.80	65.03	78.59

*. Average of five breaks unless otherwise specified.

**. After hot-plate test at 230°C. for 20 minutes.

***. After hot-plate test 240°C. for 20 minutes.

Note: 1d was treated 2 hours and 1e was treated 15 hours. A p following a treatment number denotes that the sample was padded and not washed; w denotes the treatment was washed.

Tensile strength.--The tensile strength of the fabrics before and after heating was indirectly the main factor used to determine the comparative effectiveness of the treatments. The percent strength loss was the main factor and it was determined from the tensile strength of the fabrics before and after heating. The loss in strength of the fabrics due to the action of the chemical treatments was more pronounced with the cotton fabric than either of the others. To be more specific, all the chemicals used in this research caused a loss in strength of the cotton fabric merely through their application. It was mentioned in Chapter II that cellulose is very sensitive to the effect of acids. Also, in describing the properties of the chemicals used, it was shown that most of the chemicals are weak acids. In view of these two statements it is postulated that the acidity of the chemicals used is responsible for the loss in tensile strength of the treated cotton fabrics. The average tensile strength of the treated cotton samples were all below the range of variation for the untreated fabric.

The nylon fabric proved that it is very resistant to deterioration by most organic chemicals. Only three of the chemicals used caused a loss in strength merely through their application. These chemicals were para-nitrophenol, beta-naphthol, and pyrogallol. The loss in tensile strength in some cases due to the application of the pyrogallol is not understood in view of the many treatments with this chemical which caused no loss in strength. Altogether, fifteen treat-

ments were performed using pyrogallol. Only two of these treatments indicated a loss in strength below the range of variation for the untreated fabric. A possible explanation could be in the fabric itself and not from the effect of the chemical. The beta-naphthol used was technical grade and contained many impurities. It remains to be determined whether or not pure beta-naphthol has any effect on the tensile strength of nylon. The Dacron fabric was not as inert to the action of the chemicals applied as was the nylon. The para-quinone, boric acid, para-nitrophenol, pyrogallol, and phloroglucinol all caused a loss in tensile strength merely through their application. It is again not understood why the pyrogallol and phloroglucinol caused, in some cases, a loss in strength below the range of variation for the untreated fabric. For all the treatments applied with these two chemicals, only one pyrogallol treatment and two phloroglucinol treatments caused a loss in strength below the range of variation. None of the average tensile strength values for the treated Dacron, which fell below the range of the untreated Dacron, were many pounds lower than the range. More tests with the untreated Dacron might possibly bring all the values of the treated samples within the range of the untreated fabric.

The comparative strength losses for both the different fabrics used and the different chemicals used are listed in Table 5. These strength losses were computed by the following formula: Per cent strength loss = $\frac{A - B}{A} \times 100$

Table 5. Comparative Strength Losses in Per Cent*

Treatment	After Hot-Plate Test at 230°C. for 20 min.			After Hot-Plate Test at 240°C. for 20 min.		
	Cotton	Nylon	Dacron	Cotton	Nylon	Dacron
Untreated Fabric	46.42	73.51**	13.55	64.73	93.80	53.71
Southern Mills		11.23			37.50	
Kohnstamm sample		40.34			53.33	
1d. p-Quinone	62.97	7.90	1.75	73.40	13.22	25.64
1e. p-Quinone	67.33	1.45	15.74	80.11	19.12	23.62
2. Catechol	59.28	22.31	30.66	69.83	37.61	61.09
3. Boric acid	59.96	69.72	15.86	71.28	77.48	79.19
4p. p-Nitrophenol	67.05	74.27	13.44	74.33	75.03	45.57
4w. p-Nitrophenol	36.95	17.41	0.97	58.41	54.27	30.91
5p. Pyrogallol	54.20	25.09	7.79	67.80	57.87	23.64
5w. Pyrogallol	52.28	7.10	0.12 ^a	62.18	35.22	15.42
6. Phloroglucinol	65.03	24.56	0.00	78.59	39.87	4.89
7. Phloroglucinol		15.05	1.27 ^a		18.92	7.37
8. Pyrogallol		11.75	4.32		18.73	26.35
9. Pyrogallol		8.31	6.70		30.96	19.98
10. Pyrogallol		7.66	0.23		20.35	14.43
11. Phloroglucinol		4.37	9.57		31.23	15.64

*. Average of five tests unless otherwise specified.

** . Average of twenty tests.

a. Denotes a per cent gain in strength.

Note. 1d was treated 2 hours and 1e was treated 15 hours.
The p following a treatment number denotes the sample
was padded and not washed; w following the treatment
number signifies the sample was washed.

Table 5 continued. Comparative Strength Losses in Per Cent*

Treatment	After Hot-Plate Test at 230°C. for 20 min.			After Hot-Plate test at 240°C. for 20 min.		
	Cotton	Nylon	Dacron	Cotton	Nylon	Dacron
12. Phloroglucinol		14.35	0.52		23.28	16.46
13. Phloroglucinol		8.89	3.75		25.00	11.04
14. Resin No. 1		44.39	17.46		46.57	32.16
15. <u>Beta</u> -naphthol		57.18	20.53		78.07	48.37
16. Pyrogallol		5.75	7.47			
17. Pyrogallol		7.53	7.37			
18. Pyrogallol		6.68	7.06			
19. Pyrogallol		9.93	11.98			
20a. p-Quinone		5.28			43.06	
20b. p-Quinone		7.87			16.02	
20c. p-Quinone		5.87	7.72		10.97	18.94
21a. Pyrogallol		6.53			19.20	
21b. Pyrogallol		12.13			16.13	
21c. Pyrogallol		9.89	10.88		22.56	25.05
22a. p-Quinone		6.49			17.46	
22b. p-Quinone		8.48			19.25	
22c. p-Quinone		4.08			12.23	
23a. Pyrogallol		15.65			27.80	
23b. Pyrogallol		13.53			15.83	
23c. Pyrogallol		14.59			16.31	

*. Average of five tests unless otherwise specified.

Note. The treatment numbers with a, b, and c, signify that the samples were padded one, two, and three times respectively.

where:

A = average tensile strength value for each treated sample,

B = average tensile strength value of the heated sample.

These comparative strength loss values are relative figures for comparison only and must not be construed as absolute values. An examination of Table 5 reveals that on a comparative basis none of the treatments used on the cotton fabric improved the heat resistance to an appreciable extent over the per cent strength loss of the untreated fabric. The para-nitrophenol treatment which had been washed while the fabric was still wet from the treatment gave a small decrease in the per cent strength loss as compared to the untreated fabric. This decrease in strength was not considered as any great improvement, especially in view of the fact that the para-nitrophenol treatment weakened the fabric more as a result of the treatment alone than any of the other chemicals used. This may be illustrated by reference to Table 4, treatment 4w. It is also interesting to note that the boric acid treatment used in this study failed to give any improved heat-resisting properties. As pointed out in Chapter I, the boric acid treatment is a patented heat-resisting finish for cellulosic fibers. The temperatures used in testing the fabrics in this work may have been higher than those intended for use with the treatment as patented. The boric acid decomposes at 185°C. and this could possibly be responsible for the failure of the treatment to impart any increased heat-resistance to the cotton

fabric. Continued treatments of the cotton fabric were stopped after the preliminary screening treatments revealed no improved heat-resistance with the chemicals used for this research.

The improvement of the heat-resisting properties of the nylon fabrics was considerably superior to that of the cotton fabrics. With the exception of para-nitrophenol, all the chemicals used gave some increased heat-resistance as compared with the untreated fabric when heated at 230°C. for 20 minutes. All the chemicals used improved the heat-resistance at 240°C. for 20 minutes. As listed in Table 5, the treatments of para-Quinone, pyrogallol, and phloroglucinol appeared to give the best comparative results on the nylon. The para-Quinone treatment is a United States patented treatment and was performed in this research for the purpose of comparison. The high cost of phloroglucinol has been a barrier in the development of a suitable finish with this chemical and because of this the development work has been directed toward a desirable finish using pyrogallol.

Table 6 contains information pertinent to the treatment of nylon with phloroglucinol. The various concentrations, times, and temperatures of the treatments used with this chemical are included in the table. The comparative strength losses presented for each combination which was tried are also listed. The combination of variables which gave the best results after heating at 230°C. for 20 minutes was a 0.5 per cent concentration in the bath for 30 minutes at 90°C. No

Table 6. Phloroglucinol Treatments on Nylon

Concentration (% of bath)	Time (min.)	Temp. (°C.)	Wetting Agent	Strength Loss (%)*	
				230°C. for 20 min.	240°C. for 20 min.
1.0	60	65	None	24.56	39.87
0.5	30	90	0.5% Triton X-100	15.05	18.92
0.5	30	90	None	4.37	31.23
0.5	60	90	0.1% Triton X-100	14.35	23.28
0.5	60	90	None	8.89	25.00

*. Average of five tests after heating with hot-plate tester.

wetting agent was used in this bath. The second best combination for retention of strength after being heated at 230°C. for 20 minutes was also one in which no wetting agent was used. These results were directly reversed when the fabrics were exposed to a heating of 240°C. for 20 minutes. The two samples which were treated with a wetting agent in the bath gave the best results and the bath which contained the largest per cent of the wetting agent had the smallest per cent strength loss after testing at 240°C. for 20 minutes. From the results observed in Table 6, it is concluded that at lower temperatures the amount of phloroglucinol in the fabric should be less than the amount required for higher temperatures. Therefore, the amount of phloroglucinol used for nylon should vary according

to the temperature to which the finished product will be subjected. The foregoing conclusion is based on the assumption that more phloroglucinol is absorbed by the fabric which contains the wetting agent in the bath.

The nylon treated in the bath solution of para-quinone gave excellent results after heating at both 230°C. and 240°C. for 20 minutes. The comparative strength losses of the two bath treatments are listed in Table 5, treatment numbers 1d and 1e. Other treatments of the nylon with para-quinone consisted of padding the para-quinone solution onto the nylon. The results of the padded para-quinone treatments are presented in Table 7. The strength losses were much lower with all the

Table 7. Properties of Para-quinone Padded Nylon

Sample Number	Concentration (% of bath)	Wet Pickup (%)	Dry Pickup (%)	Strength Loss (%)*	
				230°C. for 20 min.	240°C. for 20 min.
20A	0.1	59.30	0.29	5.28	43.06
20B	0.1	60.49	0.62	7.87	16.02
20C	0.1	56.56	0.86	5.87	10.97
20C**	0.1	49.80	0.61	7.72	18.94
22A	0.1	62.23	0.60	6.49	17.46
22B	0.1	55.41	0.62	8.48	19.25
22C	0.1	59.69	0.65	4.08	12.23

*. Average of five tests after heating with hot-plate tester.
 **. Dacron sample treated same as 20C nylon.

treatments of para-quinone after heating than either of the commercial fabrics tested. No trend of results could be established in the strength losses after heating at 230°C. for 20 minutes. However, after heating at 240°C. for 20 minutes the results obtained indicated that the per cent strength loss is dependent upon the amount of chemical retained by the fiber. To clarify this statement, the greatest per cent loss in strength of 43.06 occurred at the lowest dry pickup of 0.29 per cent. On the other hand, the lowest per cent loss in strength of 10.97 per cent was encountered with the largest per cent dry pickup of 0.86. With one exception the per cent strength losses at 240°C. followed this pattern.

The results of the heat-resistant properties of the nylon fabrics treated with pyrogallol in a bath solution are found in Table 8 and Table 9. Table 8 contains data on the fabrics which were washed before they were dried. Table 9 contains data on fabrics which were allowed to dry before they were washed. The fabrics which were allowed to dry before they were washed gave the best results after heating at 230°C. for 20 minutes. Therefore, it is suggested that the fabrics be allowed to dry before washing for the best results. Table 8 has one treatment listed with pyrogallol where no washing followed the bath treatment. The per cent loss in strength for this sample was very unsatisfactory and considerably higher than the losses obtained where the fabrics were washed after the treatment. These results seem to indicate that if an

Table 8. Pyrogallol Treatments on Nylon*

Concentration (% of bath)	Time (min.)	Temp. (°C.)	Wetting Agent	Strength Loss (%)**	
				230°C. for 20 min.	240°C. for 20 min.
1.0***	60	65	None	25.09	57.87
1.0	60	65	None	7.10	35.22
1.0	60	65	0.1% Triton X-100	7.66	20.35
0.5	30	90	0.5% Triton X-100	11.75	18.73
0.5	30	90	None	8.31	30.96

*. Samples washed before drying unless otherwise specified.

**. Average of five tests after heating with hot-plate tester.

***. No washing followed the chemical treatment.

Table 9. Pyrogallol Treatments on Nylon*

Concentration (% of bath)	Time (min.)	Temp. (°C.)	Wetting Agent	Strength Loss (%)**	
				230°C. for 20 min.	240°C. for 20 min.
1.0	60	65	None	5.75	
1.0	60	65	0.1% Triton X-100	9.93	
0.5	30	90	0.5% Triton X-100	7.53	
0.5	30	90	None	6.68	

*. After treating the samples were allowed to dry before washing.

**. Average of five tests after heating with hot-plate tester.

excess of the pyrogallol is allowed to remain of the nylon, excess degradation takes place. From the data presented in Table 8, the effect of the pyrogallol on the nylon fabrics follows the same pattern as the effect of the phloroglucinol on the nylon. These observations are made with the assumption that more pyrogallol is absorbed by the fabrics which are treated in a bath containing a wetting agent also. After being heated at 230°C. for 20 minutes, the nylon which was treated with no wetting agent gave the best results. At 240°C. for 20 minutes, the nylon which had been treated with a wetting agent gave the best results and the sample which was treated in the bath with the largest concentration of the wetting agent gave the best results. This is identical with the action of phloroglucinol on the nylon. It is worth repeating at this time that a larger amount of pyrogallol is needed in the fabric which is to be subjected to the higher temperatures. For lower temperatures, a smaller amount of the pyrogallol on the fabric gives better results.

No trend could be determined on the nylon which was padded with a pyrogallol solution. The fabrics which were treated in a solution containing a wetting agent gave better heat-resisting properties than samples padded identically with no wetting agent in the bath.

The experimental chemicals used in this research not only gave good heat-resisting properties to the nylon fabrics, they imparted good heat-resisting properties to the Dacron

Table 10. Properties of Pyrogallol Padded Nylon

Sample Number	Concentration (% of bath)	Wet Pickup (%)	Dry Pickup (%)	Strength Loss (%)*	
				230°C. for 20 min.	240°C. for 20 min.
20A**	0.1	48.13	0.31	6.53	19.20
21B**	0.1	48.78	0.31	12.13	16.13
21C**	0.1	51.54	0.62	9.89	22.56
21C***	0.1	46.64	0.42	10.88	25.05
23A	0.1	54.38	0.31	15.65	27.80
23B	0.1	53.05	0.36	13.53	15.83
23C	0.1	55.10	0.58	14.59	16.31

*. Average of five tests after heating with hot-plate tester.

**. 0.1 per cent Triton X100 wetting agent used with these samples.

***. Dacron sample treated same as 21C nylon.

fabrics tested also. The per cent strength loss was decreased more on the treated nylon samples, rather than the Dacron samples, and for this reason the development work was mainly limited to nylon fabrics after the screening tests had been performed. The results of all Dacron treatments are found in Table 3. The screening tests on Dacron indicated that phloroglucinol and pyrogallol were the best two experimental chemicals used for imparting heat-resistance to the Dacron fiber. Table 11 contains the results of Dacron treated by a bath process with various concentrations of phloroglucinol. The results appear to be very good and attention is called to the sample

Table 11. Phloroglucinol Treatments on Dacron

Concentration (% of bath)	Time (min.)	Temp. (°C.)	Wetting Agent	Strength Loss (%)*	
				230°C. for 20 min.	240°C. for 20 min.
1.0	60	65	None	0.00	4.89
0.5	30	90	0.5% Triton X-100	1.27**	7.37
0.5	30	90	None	9.57	15.64
0.5	60	90	0.1% Triton X-100	0.52	16.46
0.5	60	90	None	3.75	11.04

*. Average of 5 tests after heating with hot-plate tester.

**. Denotes a gain in strength.

which had a gain in strength due to the treatment. Some of the Dacron samples contracted on heating and this is believed to be partially the cause for the strength gain of the Dacron sample. The treatment undoubtedly aided in this phenomenon also, because it must be remembered that the raw Dacron also underwent a contraction due to the heat. A close observation of the results confirms the fact that the samples which absorbed the largest amount of the chemical are the ones which possess the better heat-resisting properties as determined with the hot-plate tester.

The Dacron samples which were treated by a bath process with pyrogallol also gave very good results. Here again one treatment caused a slight increase in strength of the fabric tested. Table 12 contains all information on the Dacron

Table 12. Pyrogallol Treatments on Dacron*

Concentration (% of bath)	Time (min.)	Temp. (°C.)	Wetting Agent	Strength Loss (%)**	
				230°C. for 20 min.	240°C. for 20 min.
1.0***	60	65	None	7.79	23.64
1.0	60	65	None	0.12****	15.42
1.0	60	65	0.1% Triton X-100	0.23	14.43
0.5	30	90	0.5% Triton X-100	4.32	26.35
0.5	30	90	None	6.70	19.98

*. After treating the samples were washed before drying unless otherwise specified.

**. Average of five tests after heating with hot-plate tester.

***. Sample was run through the padder to remove excess solution and no washing followed treatment.

****. Denotes a gain in strength.

Table 13. Pyrogallol Treatments on Dacron*

Concentration (% of bath)	Time (min.)	Temp. (°C.)	Wetting Agent	Strength loss (%)**	
				230°C. for 20 min.	240°C. for 20 min.
1.0	60	65	None	7.47	
1.0	60	65	0.1% Triton X-100	11.98	
0.5	30	90	0.5% Triton X-100	7.37	
0.5	30	90	None	7.06	

*. After treating the samples were allowed to dry before washing.

**. Average of five tests after heating with hot plate tester.

treated with pyrogallol and washed before allowing the treated samples to dry. Table 13 contains the information of identical treatments with the exception that the fabrics were allowed to dry after the treatment before washing. The data obtained in these two tables tends to show that better heat-resisting properties with Dacron are obtained if the samples are washed before they are allowed to dry. This would seem to indicate that any excess pyrogallol which did not react with the fiber should be removed for the best results. Since phenol reacts with Dacron by hydrogen bonding, it could be theorized that pyrogallol reacts in the same manner. Another possible source of attraction could be Van der Waals forces. Dacron padded with pyrogallol, Table 10--number 21C, failed to have any outstanding heat-resisting properties. The per cent strength loss after heating was only a few points lower than the per cent strength loss for the untreated Dacron. This would indicate that for a treatment to be effective on Dacron, the fabric must remain in the solution for a period of time which is longer than the time required to pad the fabric. This longer time of treatment appears to be necessary in order for the chemical to penetrate the Dacron fiber in order for the chemical reaction to take place.

Color and hand of the treated samples.--The experimental chemicals affected the color of the fabrics in many varied ways. Tables 14, 15, and 16 list the various color changes produced in the fabrics by the treatment alone and also the color

Table 14. Effect of Chemical Treatment on Color of Fabric*

Treatment	Cotton	Nylon	Dacron
Untreated Fabric	White	White	White
Southern Mills Pyron H Sample	-----	Purple	-----
Kohnstamm Sample	-----	Orange Brown	-----
p-Quinone	Brown tint	Reddish Brown	Brown Tint
Catechol	No Change	Pink Tint	No Change
Boric Acid	No Change	No Change	No Change
p-Nitrophenol	No Change	Bright Yellow	No Change
Pyrogallol	No Change	Beige	No Change
Phloroglucinol	No Change	Light Beige	No Change
Resin No. 1	-----	Yellow Brown	Medium Brown
B-Napthol	-----	Brown Tint	No Change**

*. Color of the fabric after treating with the chemical before the hot-plate test.

**. After five weeks the color turned a yellowish brown.

Table 15. Effect of 230°C. for 20 Minutes on Color of Fabrics*

Treatment	Cotton	Nylon	Dacron
Untreated Fabric	Medium Brown	Medium Brown	No Change
Southern Mills Pyron H Sample	-----	Very Dark Brown	-----
Kohnstamm Sample	-----	Dark Brown	-----
p-Quinone	Light Brown	Medium Brown	No Change
Catechol	Medium Brown	Dark Brown	Light Brown
Boric Acid	Light Brown	Medium Brown	No Change
p-Nitrophenol	Light Brown	Dark Brown	No Change
Pyrogallol	Light Brown	Medium Brown	Gray Tint
Phloroglucinol	Light Brown	Medium Brown	Light Brown
Resin No. 1	-----	Dark Brown	Dark Purple
B-Naphthol	-----	Medium Brown	Light Brown

*. Fabrics heated with a hot-plate tester devised for that purpose.

Table 16. Effect of 240°C. for 20 Minutes on Color of Fabrics*

Treatment	Cotton	Nylon	Dacron
Untreated Fabric	Dark Brown	Dark Brown	Light Brown
Southern Mills Pyron H Sample	-----	Very Dark Brown	-----
Kohnstamm Sample	-----	Very Dark Brown	-----
p-Quinone	Light Brown	Dark Brown	Light Brown
Catechol	Medium Brown	Very Dark Brown	Light Brown
Boric Acid	Light Brown	Dark Brown	No Change
p-Nitrophenol	Medium Brown	Very dark Brown	Light Brown
Pyrogallol	Medium Brown	Dark Brown	Gray Tint
Phloroglucinol	Light Brown	Dark Brown	Light Brown
Resin No. 1	-----	Dark Brown	Dark Purple
B-Naphthol	-----	Dark Brown	Light Brown

*. Fabrics heated with a hot-plate tester devised for that purpose.

changes produced after the application of heat to the samples. The change in color produced by some of the chemicals on the fabrics seemed to indicate a chemical reaction with the fabrics. If this is true, the treatments which produce a color change in the fabrics should be of a permanent nature. After heating some of the nylon treated samples, the color of the samples was very dark or scorched. The strength losses indicated that the scorching failed to cause any appreciable degradation to the fabrics. Some of the samples which appeared to be scorched to a high degree had a very small loss in strength. Under these conditions it could be said that the serious change in color could be due to the discoloration of the chemicals used and not a tendering of the fabric.

Tables 17, 18, and 19 list the changes in the hand of the fabrics after the chemical treatments and also after the treated samples were heated. It should be pointed out that the better treatments for the nylon produced a slight harsh feel to the fabric. The resilience of the fabric was not impaired but the fabric appeared to have a layer of the chemical on its surface which caused the harsh feel. This harshness was very slight and would not subtract from the better qualities of the finished fabric. After the fabrics were heated the experimental treatments became slightly stiff after 230°C . for 20 minutes and stiff after 240°C . for 20 minutes. The commercial fabrics tested at the same time were considerably stiffer after the heat test than the pyrogallol and

Phloroglucinol Treated fabrics. The Dacron samples seemed to fuse as a result of the heat test and upon cooling they became very stiff and resilient.

Table 17. Effect of Chemical Treatment on Hand of Fabrics*

Treatment	Cotton	Nylon	Dacron
Southern Mills Pyron H Sample	-----	Stiff	-----
Kohnstamm Sample	-----	Stiff	-----
p-Quinone	No Change	Harsh	Harsh
Catechol	No Change	No Change	Harsh
Boric Acid	Harsh	No Change	No Change
p-Nitrophenol	No Change	Oily Feel	Harsh
Pyrogallol	Harsh	Harsh	No Change
Phloroglucinol	No Change	Harsh	No Change
Resin No. 1	-----	Stiff	Stiff
B-Naphthol	-----	Harsh	Very Harsh

*. After the chemical treatment before heating with the hot-plate tester.

Table 18. Effect of 230°C. for 20 Minutes on Hand of Fabrics*

Treatment	Cotton	Nylon	Dacron
Untreated Fabric	No Change	No Change	Stiff
Southern Mills Pyron H. Sample	-----	Very Stiff	-----
Kohnstamm Sample	-----	Very Stiff	-----
p-Quinone	No Change	Slight Stiffness	Stiff
Catechol	No Change	Stiff	Very Stiff
Boric Acid	Harsh	Little Change	Stiff
p-Nitrophenol	No Change	Very Stiff	Very Stiff
Pyrogallol	Harsh	Slight Stiffness	Very Stiff
Phloroglucinol	No Change	Slight Stiffness	Very Stiff
Resin No. 1	-----	Very Stiff	Very Stiff
B-Naphthol	-----	Harsh, Stiff	Very Stiff

*. Fabrics heated with a hot-plate tester devised for that purpose.

Table 19. Effect of 240°C. for 20 Minutes on Hand of Fabric*

Treatment	Cotton	Nylon	Dacron
Untreated Fabric	No Change	Stiff	Very Stiff
Southern Mills Pyron H Sample	-----	Very Stiff	-----
Kohnstamm Sample	-----	Very Stiff	-----
p-Quinone	No Change	Stiff	Very Stiff
Catechol	Harsh	Very Stiff	Very Stiff
Boric Acid	Harsh, Stiff	Slight Stiffness	Very Stiff
p-Nitrophenol	No Change	Very Stiff	Very Stiff
Pyrogallol	Harsh	Stiff	Very Stiff
Phloroglucinol	Harsh	Stiff	Very Stiff
Resin No. 1	-----	Very Stiff	Very Stiff
B-Naphthol	-----	Harsh, Stiff	Very Stiff

*. Fabrics heated with a hot-plate tester devised for that purpose.

CHAPTER VI

CONCLUSIONS

The conclusions shown below are based on the results obtained in this study for the chemicals and conditions specified. They should not be construed as holding for any other situations.

The heat-resistance of cotton fabric is lowered as a result of the application of the experimental chemicals. All chemicals employed in this study caused a decrease in the heat-resistance of the cotton fabric. For this reason it is concluded that phenolic structures of the type employed in this study will not impart increased heat-resistance to cotton fabrics.

All chemicals employed in this study produced increased heat-resistance of the nylon fabric tested with the exception of para-nitrophenol. Phenolic structures of the type used may be said to impart increased heat-resistance to nylon.

Catechol, para-quinone, boric acid, resin number 1, and beta-naphthol caused a decrease in the heat-resistance of Dacron after heating at 230°C. for 20 minutes. Only catechol and boric acid caused a decrease after heating at 240°C. for 20 minutes.

Pyrogallol and phloroglucinol imparted greater heat-resistance to nylon and Dacron than the other chemicals tested.

On the basis of the cost of the chemical, pyrogallol would be recommended rather than phloroglucinol. The treatments with the two chemicals produce an increased heat-resistance of approximately the same magnitude on a nylon fabric. Therefore, it is concluded that a heat-resisting finish has been developed in the laboratory which has a commercially feasible finishing formulation. Subject to confirmation by service tests, it is believed that this finish will yield a product favorably competitive with the better products presently available.

CHAPTER VII

RECOMMENDATIONS

From the promising results obtained with the phenolic compounds used in this study, a continuation of experiments using other chemicals with a similar structure would appear to give suitable results also. Therefore, it is suggested that continued work be performed with phenolic compounds of the type used herein. This study should include the double and triple ring phenolic compounds.

There is very little information on a few of the fibers as to what happens inside them when they are subjected to heat. This is especially true of Dacron and nylon. It is suggested that more research be directed toward the investigation of the effect of heat on the breaking of the polymer and why the fibers lose strength as a result of high temperatures.

The chemicals employed in this study gave a good heat-resisting finish as determined by laboratory tests. Therefore, it would be interesting to determine the comparative effectiveness of the treatments under actual service conditions. This could be accomplished by preparing several laundry press covers from material treated according to the specifications listed in this work and distributing them for use in laundries for comparison with present covers.

A study to determine the possible bonding sites of the chemical to the fiber should also prove interesting and informative. A possible way in which this might be accomplished would be through a special spectrophotometric process.

An alteration of the formulation as presented herein might also produce a better heat-resistant finish than the ones obtained by this study. A suggestion toward this end would be the binding of the chemicals to the fabric through the use of a resin medium or possibly a combination of two or more of the chemicals which gave the better results.

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