To the Graduate Council:

I am submitting herewith a thesis written by Betsy Carolyn Westlake entitled "The Effect of Selected Reactive Dyes on the Flammability Behavior of Cotton." I recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Textiles and Clothing.

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THE EFFECT OF SELECTED REACTIVE DYES ON THE FLAMMABILITY BEHAVIOR OF COTTON

A Thesis

Presented for the

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Degree

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Betsy Carolyn Westlake

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ABSTRACT

The flammability behavior of reactive dyed cotton fabrics was investigated, and the results were compared to the burning behavior exhibited by undyed cotton fabric. A 2 x 3 x 2 factorial design was used to evaluate the effects of dye type, dye color, and dye level on the peak heat transfer rates, time to reach the peak heat transfer rates, total burn time, total heat transfer, heat/time relationship, rate of flame spread, and heat of decomposition of the dyed fabrics. Reactive dyed cotton fabrics included two dye types (Procion H and MX), three dye colors (red, yellow, and blue) and two dye levels (1% and 4%). Testing was carried out on the Mushroom Apparel Flammability Tester (MAFT), 45° angle tester, and Differential Scanning Calorimeter (DSC). In addition, undyed cotton fabric was tested for comparison with the dyed fabric.

The MAFT findings indicated that dye level is the most important factor in the heat transfer rate at the second peak, the time to reach the second heat transfer rate peak, and total heat transfer of the dyed fabrics. No differences were found in the heat transfer rate at the first peak, the time to reach the first heat transfer rate peak, and total burn time for the 13 dyed and undyed fabrics.

The effect of dye level was observed as an increase in the values for the heat transfer rate at the second peak, the time to reach the second heat transfer rate peak, and total heat transfer. The undyed fabric exhibited lower values than the 4% dyed fabrics for these variables but was not significantly different from the 1% dyed fabrics.

Heat/time relationship analysis indicated the fabrics had a slow initial rate of heat transfer followed by a rapid acceleration in the rate and finally a leveling off as the total amount of heat transferred by the fabric was reached. The analysis revealed no difference in the flammability hazard of these fabrics during the first minute of burning.

Only dye level affected the 45° angle test measurement of rate of flame spread for the dyed fabrics, with the 4% fabrics exhibiting the longest flame spread times. Dyed and undyed fabrics exhibited no difference in rate of flame spread.

DSC results revealed that both the chemical structure of the dye, including the dye chromophore, and the amount of dye are significant in the decomposition of reactive dyed cellulose. The effect of dye level was observed as a decrease in the values for the temperature at onset of degradation, the degradation peak temperatures, and the peak heights ratio. Dye color was significant for heat of decomposition, the temperature at onset of degradation and the degradation peak temperatures. Heat of decomposition differences were related to dye type and dye level. Yellow fabrics remained relatively constant across dye type and dye level while the red and blue fabrics exhibited contrasting increases and decreases across dye type and dye level.

DSC results for the undyed fabric indicated the fabric begins to degrade at a higher temperature, exhibits only one degradation peak, exhibits a higher degradation peak temperature, and requires less energy for decomposition than the dyed fabrics. Thus, the second degradation peak for the dyed fabrics may represent the decomposition of portions of the cellulose molecule containing dye molecules.

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CHAPTER I

INTRODUCTION

Textile fabrics may constitute a fire hazard due to their susceptibility to ignition and decomposition under favorable conditions (28). An awareness of the tragic burn injuries caused by the flammability of textile materials has resulted in considerable protective legislation, federal standards, and textile flammability research over the past 30 years (4). Efforts have been aimed at providing the public maximum protection against the flammability hazards of textiles. The physical and mental anguish which burn injuries create is the major justification for federal action and research, although the magnitude of the problem has been difficult to identify. Early burn statistics from the U.S. Department of Health estimated that fabric flammability caused 5,000 deaths and up to 250,000 injuries annually (12). More recent data show more conservative estimates of 517 deaths and 15,600 injuries (27). Overall, textile flammability accounts for 20% of the total loss of life and property due to fires of all types (28).

A series of tragedies in the 1940s and early 1950s involving the flash burning of brushed rayon "torch" sweaters and children's cowboy chaps initiated congressional action in the area of fabric flammability and led to passage of the Flammable Fabrics Act (FFA) in 1953, prohibiting the introduction or movement in interstate commerce of highly flammable apparel and fabrics (4,26,27). In 1967 this cornerstone of fabric flammability legislation was amended to give the Secretary of Commerce broader authority in promulgating specific standards to protect the public against unreasonable risk and to extend coverage to interior furnishings (4,26). In 1972 the U.S. Consumer Product Safety Commission (CPSC) was created and given jurisdiction over product safety including the transferral of FFA responsibilities to the CPSC (26).

Since 1953 three federal standards for apparel textiles have been promulgated to implement the FFA. Title 16 CFR 1610, passed in 1953, provides a method for rating the flammability of textiles for clothing use by a 45° angle ignition and rate of burn test (21,26). This standard has successfully removed dangerously flammable fabrics from the market. The two additional standards, Title 16 CFR 1615 and Title 16 CFR 1616, cover children's sleepwear and were passed in 1971 to include sizes 0-6x and in 1974 to include sizes 7-14, respectively. A considerably more stringent vertical test is used for these standards (21,26), which have effectively reduced the number of sleepwear related burn injuries. In 1975 Title 16 CFR 1615 had been in effect for three years and sleepwear related burns had declined from 14% to 7% of the total clothing related burns. The added protection of Title 16 CFR 1616 over the next two years brought the estimated rate down to 3% in 1977 (53).

Since implementation of the sleepwear standards the CPSC has enlisted the help of both industry and the National Bureau of Standards (NBS) to work on expanding flammability coverage to general wearing apparel. In 1975 the CPSC proposed a schedule for extending

Title 16 CFR 1616 to six categories of apparel. The schedule's compliance dates for the regulation were as follows: women's nightgowns--Sept. 1, 1976, adult women's robes and housecoats--1978, adult pajamas--1979, dresses for all ages--1980, men's shirts--1981, and men's and boy's pants--1981 (32). However, this schedule has not been adopted, and progress in the area of apparel flammability has been slow due to experiences with current flammability standards, problems inherent to this area of flammability, and current research findings.

The limitations of the current flammability standards were quickly recognized. Evaluation of the fire hazard of a material and simulation of real-life clothing ignition cannot be adequately accomplished using the existing standard test methods (12). Thus, the primary thrust by the CPSC, NBS, and industry over the past five years has been to identify reasonable test methods which relate to potential burn hazard (58) and characterize the flammability behavior of fabrics and garments.

Identification of reasonable test methods first involved the determination of the fabric and garment properties which contribute to flammability hazard. The following properties have been identified: (a) ease of ignition, (b) rate of flame spread, (c) rate of heat release or transfer, (d) total amount of heat released or transferred, (e) duration of burning or ease of extinguishment, and (f) garment configuration (7,8,34). The interaction or relationship between properties such as rate of heat transfer and the duration of burning can be significant. For example, while little or no injury results from exposure to a large amount of energy produced over an extended period of time, exposure to an equal amount of energy released instantaneously may result in serious damage (7).

NBS's work in this area resulted in the development of the Proposed Standard for the Flammability of General Wearing Apparel, submitted to the CPSC in 1976. In developing this standard NBS established a new test method and testing concept which characterize fabrics according to hazard. The method includes the use of an apparatus called the Mushroom Apparel Flammability Tester (MAFT). The MAFT simulates the burning of a garment by suspending a vertical cylindrical specimen from the closed top plate of a vertical cylinder which embodies heat sensors. The specimen is ignited in the body of the fabric to allow flame spread in all directions. Ease of ignition and rate of heat transfer are measured in separate experiments and are used to classify the fabric. The test concept also includes a garment classification system which requires the use of safe fabrics in hazardous, loose fitting garments and allows the use of fabrics with greater injury potential in less hazardous, tight fitting garments (30,46).

The proposed standard, however, has not been adopted, and even though the MAFT continues to be critically evaluated and used as a research tool to characterize the flammability behavior of fabrics, more research is needed in order for the CPSC to deal appropriately with apparel flammability. While the importance of the chemical composition of textiles (i.e., the chemical structure of the fiber or fibers, impurities, and additives) in determining flammability behavior (54) and the significance of hazard properties are recognized, current research has not covered these areas fully. Considerable research has been conducted to identify hazard properties of fabrics and to evaluate the influence of fiber content, fabric weight, flame retardants, and fabric layers on these hazard properties; however, very little attention has been paid to chemical additives such as dyes.

Obviously the majority of fabrics available for consumer use contain dyes. Early studies using thermogravimetric analysis indicate that dyestuffs do influence the combustion and pyrolysis of dyed fabrics (50). More recently, a brief look at heat transfer and ignition of dyed fabrics showed small differences between dyed and undyed fabrics (24) but did not provide a detailed look at the ways dyestuffs alter the combustion of dyed fabrics. Heat transfer data can provide more complete information about fabric flammability behavior and hazard if the data are analyzed in conjunction with burn time and if information is obtained from more than one test instrument. Thus, a detailed analysis of heat/time relationships using more than one test instrument may provide valuable information about the effects of dyes on fabric flammability.

Certainly the need for flammability dye information is most important for fabrics which do burn readily and do represent a hazard. Flammable Fabrics Accident Case and Testing System's 1973 data base indicates that 100% cotton fabric is the most frequently ignited fabric and that cellulosics generally produce more serious burns than do thermoplastics (57). Additional 1973 data show that 100% cellulosic fabrics and cellulosic blend fabrics accounted for 66.7% of the garment production and 93.7% of the reported burn cases and that 100% synthetic fabrics accounted for 32.5% of the garment production and only 5.3% of the reported burn cases (51).

Moreover, of the four dye classes used on cotton fabric, reactive dyes require more study than do direct, vat, and sulfur dyes. Reactive dyes combine chemically with the hydroxyl groups on the cellulose molecule (43) while direct, vat, and sulfur dyes are physically absorbed by the fabric. Previous research indicates that chemically bound dyes may be more likely to change the combustion and pyrolysis pattern of the fabric than a dye which is physically held on the fabric (50).

Since more quantitative and detailed data on heat transfer from burning fabrics in relation to burn injury are needed (29), as well as information on the flammability of dyed fabric, this study was designed to evaluate the influence of selected reactive dyes on the flammability behavior of cotton fabric through an analysis of heat and time data obtained from the MAFT, burning rates data from the 45° angle tester, and heat of decomposition information from the Differential Scanning Calorimeter.

I. OBJECTIVES

1. To determine the effect of dye type, dye color, and dye level on the peak heat transfer rates, time to reach the peak heat transfer rates, total heat transfer, total burn time, heat/time relationship, rate of flame spread, and heat of decomposition of reactive dyed cotton fabric.

2. To determine the difference between undyed and reactive dyed cotton fabric in peak heat transfer rates, time to reach peak heat transfer rates, total heat transfer, total burn time, heat/time relationship, rate of flame spread, and heat of decomposition.

II. ASSUMPTIONS

1. Knowledge of fabric flammability can be acquired by modeling real-life situations and the results of such experiments may increase understanding of actual clothing-related burn incidents.

2. The fiber-reactive dyes used in this study are representative of those used on cotton fabric commercially.

III. LIMITATIONS

1. Fabrics of 100% cotton will be the only fabric tested.

2. Only plain weave fabric of one weight will be used.

3. Only six fiber-reactive dyes representing two types and three colors will be used.

4. Fabrics will be dyed at only two levels.

5. All flammability testing will be carried out on three instruments: the MAFT, the 45° angle tester, and the DSC.

IV. DEFINITIONS

Differential Scanning Calorimeter (DSC)--An apparatus which measures the heat energy per unit time necessary to maintain a sample and reference material at the same temperature during an interval of programmed temperature rise.

Dye color--The color which results from the chromophore attached to the chemical structure of the dye, specified as either red, yellow, or blue.

Dye level--Amount of dye on the fabric expressed as a percentage of fabric weight.

Flammability--Ability to support combustion.

Heat/time relationship--Total amount of heat (in joules/cm^2) which a fabric specimen transfers to the MAFT apparatus as a function of time (calculated for every 12 seconds).

Heat of decomposition--Energy (in calories/gram) obtained by Differential Scanning Calorimetry.

Heat transfer rate--Rate at which heat is transferred from a burning specimen to the MAFT test apparatus, measured in joules/cm^2/second.

Ignition source--Flame produced from the gas, burner, and gas flow rate as specified in the procedure (46).

Mushroom Apparel Flammability Tester (MAFT)--The apparatus developed by the National Bureau of Standards for use in a general flammability standard. The MAFT is designed to measure ignition time and heat transfer rate.

Rate of flame spread--Time required for a specimen ignited according to the 45° angle test method to burn the stop-cord.

Sample--Total number of specimens required per replication for a test method.

Specimen--Dimensions of a fabric section ready for testing as specified for a test method.

Total burn time--Number of seconds during which the fabric specimen burns in the MAFT test apparatus.

Total heat transfer--Total amount of heat transferred from a burning specimen to the MAFT test apparatus.

45° angle tester--The apparatus which measures rate of flame spread and is used as the test method for Title 16 CFR 1610.

CHAPTER II

REVIEW OF THE LITERATURE

Recent literature pertaining to the broad subject of textile flammability was reviewed under the following headings: Evaluation of Flammable Fabric Hazard, Effect of Dyes on Flammability Behavior, Research Using the Mushroom Apparel Flammability Tester, and Cellulose Decomposition and Combustion.

I. EVALUATION OF FLAMMABLE FABRIC HAZARD

Burn injuries involving apparel fabrics result from the interaction of environmental, human, and physical factors (9). The importance of characterizing these factors in order to adequately evaluate the hazard of flammable fabrics has been recognized and investigated by several researchers (9,33,37,38,59). Much of this research has involved an analysis of fire accident cases from data bases such as the National Bureau of Standard's Flammable Fabrics Accident Case and Testing System. Results indicate that the severity of a burn injury depends on the circumstances surrounding the fire incident (i.e., the activities leading up to the accident, the nature of the accident, the availability of help, and the reaction of the victim) (9,59).

Buchbinder (9) has identified three classes of activities leading to apparel fires as situations in which the victim was "using fire," "playing with an ignition source," or "using flammable liquids." These activity patterns are related to the age and sex of the victim.

Age and sex are also important in the victim's reaction or defense capabilities. Buchbinder (10) found the most frequent reaction of people involved in apparel fires is running, which may increase burning. Other common reactions, in order of frequency, include beating flames with hands, removing clothing, and dropping to the ground and rolling.

The nature of the fire incident also is related to the classes of activities identified since flammable liquids are involved in approximately 40% of the burn injury cases (9,37,59). A comparison of injuries sustained from flammable fabrics with and without the involvement of flammable liquids reveals that the absence of flammable liquids results in a high percentage of minor injuries, a relatively small percentage of moderately severe injuries, and a relatively high percentage of severe injuries while flammable liquid fire incidents result in fewer minor injuries, more moderately serious injuries, and fewer severe injuries (9). In addition, running was the most frequent reaction when no flammable liquids were involved, and rolling on the ground was the most prevalent reaction in a flammable liquid accident (10).

The fabric or fabrics involved in the fire incident play an important role in the nature of the accident and the resulting burn injury. The area and depth of injury are determined by the amount and rate of heat absorption by the skin (29). Extensive studies of the physiology of burns show that the mechanism by which burns are produced depends on the time rate of heat exposure and not simply on the duration of exposure (7). Prognosis in a thermal injury is dependent on the percentage of the total skin area subjected to second and third degree burns in relation to the age and physical condition of the victim (9,29).

The threshold temperature for skin damage is 44°C. Injury occurs continuously when the surface skin temperature is above 44°C, and the rate of injury increases logarithmically with a linear increase in temperature. Thus damage occurs 100 times faster at 50°C than at 45°C. Irreversible skin damage occurs at a surface skin temperature of 72°C (1,52). The heat flux of most burning fabrics is from 0.3 to 1.0 cal/cm^2/sec, which exceeds the skin's tolerance level (6,13). An irradiance level of as low as 0.1 cal/cm^2/sec produces skin temperatures above 44°C (1).

Fabrics reach these injurious levels of heat transfer when the fabric ignites and continues to burn after ignition. Ignition, flame propagation, and heat transfer are affected by variables such as fiber composition, fabric weight, garment configuration, and the number and spatial arrangement of layers. Laboratory research has contributed information, beyond what an analysis of burn injury cases shows, which characterizes the effects of these variables on the hazard potential of flammable fabrics.

Fiber content of fabrics is important in determining flammability behavior. Fabrics composed of synthetic thermoplastic fibers melt, shrink, drip, and are not easily ignited (17,29). If ignition occurs, the flame spreads slowly and the fabrics often self-extinguish. Wool fabrics behave in a similar manner (i.e., flame propagation is slow and wool often extinguishes itself). Cellulosic fabrics, on the other hand, have no inherent self-extinguishing mechanism and will generally burn until the fabric is destroyed (13). Logically, synthetic fabrics generally produce less heat than cellulosic fabrics (40). This is consistent with burn injury data which show that cellulosic fabrics produce more serious burns and are involved in a greater proportion of burn injuries than synthetic fabrics (51,57).

Many fabrics are blends of two or more fibers. The flammability behavior of blends is influenced by the nature and proportion of the component fibers and often exceeds that anticipated from the behavior of the individual components (28,35,41). Many thermoplastic and non-melting fiber blend fabrics are more flammable than fabrics of thermo-plastic fibers alone due to what is known as the scaffold effect. For example, in a cotton/polyester blend the cotton forms a carbonaceous grid during burning which prevents the polyester from melting away from the heat source (44). Wool/polyester blends also behave more severely than the components alone as a result of interactions that occur during polymer decomposition (35).

The effect of fabric weight on ignition, flame propagation, and heat transfer has been characterized primarily for cellulosic fabrics. Ignition time and total heat transfer are proportional to fabric weight, whereas rate of flame spread is inversely proportional to fabric weight (7,17). Thus, heavy fabrics transfer more heat but are more difficult to ignite and take longer to reach high heat levels than lightweight fabrics (28,29).

The degree of garment fit and the amount of the body covered have been found to influence the severity of burn injuries. Loose fitting garments are more hazardous than close fitting garments since the fabric-skin distance can allow ample air supply for flame propagation, while close fabric-skin contact can act as a fire stop. In addition, garments which cover more than half the body generally result in more severe burn injuries (11).

The number and spatial arrangement of fabric layers greatly influence flammability behavior. Chouinaird et al. (13), using a heat sensing board, found the heat transfer from double layers of cotton or cotton/polyester fabrics was equal to the sum of the heats transferred by the fabrics singly. In addition, heat transfer from thermoplastic/cotton layers depends on which fabric is on top. Heat transfer was greatest when cotton was closest to the sensors. Finkel and Block (16) tested double layers of fabrics used in women's night-gowns and robes. Results indicated that combinations such as acetate-nylon/brushed acrylic burned more severely than would be expected from the burning of individual fabrics, while a combination of cotton flannel/cotton duster was less hazardous when burned in combination than as single layers. Shirt/undershirt assemblies were evaluated by McCullough (36). These assemblies exhibited a synergistic effect (i.e., the flammability hazard associated with the assembly was greater than the sum of the single layers would suggest). In addition, an increase in spatial separation between the layers resulted in an increase in ignition time and a decrease in heat transfer.

II. EFFECT OF DYES ON FLAMMABILITY BEHAVIOR

The majority of fabrics on the market contain dyes. Most of the research conducted to characterize the flammability behavior of different fibers has involved undyed fabrics in an effort to eliminate dye interference. The few studies conducted comparing dyed and undyed fabrics indicate dyes may influence the combustion and pyrolysis of the material.

Early studies using dyes were initiated by an interest in utilizing dyes as a different method for obtaining flame retardancy in cotton and cotton/polyester fabrics (50,56). Segal et al. (50,56) used oxygen index (OI) and thermogravimetric analysis (TGA) to evaluate the effect of vat, ingrain, direct and reactive dyes on cotton. The vat dyes chosen were indigo dyes which contain bromine, an element known for its flame retardancy properties. Indigo dyes reduced OI values at low dye add-on levels (<1%); however, higher add-on levels increased OI values. In addition, lower decomposition temperatures were obtained as dye add-on increased. The presence of halogens (Cl, Br) in the dye was found to have very little effect on OI values; however, as halogen substitution increased, TGA curves were shifted to lower decomposition temperatures. The amount of char remaining after the completion of decomposition increased with increasing add-on (56).

Segal et al. (50) also tested phthalocyanine dyes, including one each of ingrain, vat, and direct dyes. Phthalocyanine dyes have a high nitrogen content and contain metal ions (copper, cobalt, or nickel), thus reduced flammability was expected for these dyes. However, the ingrain dye containing copper and the vat dye containing cobalt both lowered OI while the direct dye containing copper and sulfonyl groups (not in the other two dyes) raised OI.

The direct and reactive (Procion class) dyes evaluated by Segal et al. (50) showed different effects on OI. The direct dyes raised OI whereas the reactive dyes exhibited only a slight and inconsistent effect on OI. Two of the reactive dyes contained copper; however, the presence or absence of copper did not have any definite influence on the direction taken by OI. Both direct and reactive dyes had a considerable effect on pyrolysis. The greatest change was seen with the reactive dyes. Reactive dyes resulted in earlier onset and completion of decomposition and an increase in residue. TGA curves also indicated that the copper containing reactive dyes lowered the onset of decomposition more than dyes which did not contain copper.

The shifts in TGA curves caused by the reactive dyes were similar to those found for acid-dyed aminized cotton, also tested by Segal et al. (50). Their results confirmed the findings Reeves, McMillan, and Guthrie obtained in 1953. The similarity of the reactive-dyed cotton and the acid-dyed aminized cotton clarified the effect of dye-induced changes in cotton structure on cellulose decomposition. Reactive dyes react with the cellulose molecule to form cellulose derivatives in which the dye becomes a substituent group bound by covalent bonds, thus interfering with the pyrolysis of cotton to levoglucosan. In acid-dyed aminized cotton the acid dye becomes a substituent through salt bonding to the amino group and results in a similar change in pyrolysis (50).

Hopkins and Buchanan (24) studied the effect of two reactive dyes, a monochlorotriazine and a dichlorotriazine, on the flammability of cotton, cotton/polyester, and polyester fabrics using OI and the

MAFT. Ignition time, to the nearest tenth of a second, and maximum heat transfer rate were measured on the MAFT. Results indicated a trend toward decreasing OI with increasing dye level (up to 2%). Ignition time showed a statistically significant decrease with increasing dye level; however, none of the differences were more than a few tenths of a second. Dye level exhibited no effect on maximum heat transfer rate.

The effect of disperse dyes on the flammability behavior of polyester has also been studied (15). Ease of ignition and heat emission were measured using the MAFT and a Textile Research Institute (TRI) convection calorimeter, respectively. An expanded version of the vertical test used for Title 16 CFR 1615 and 1616 was used to measure residual flame time, specimen after flame, char length, and burn time. Both ease of ignition and heat emission appeared to be more sensitive to the influence of dyes than the other three measures. Dye add-on level was not a significant factor even though dye significantly affected flammability behavior. Disperse azo dyes had the most effect while quinophthalone and anthraquinone dyes exhibited little effect.

A study similar to the polyester/disperse dye study was carried out using acid dyed nylon 6 (43). The same instruments were employed with the addition of the Motor Vehicle Safety Standard No. 302, a horizontal test which measures burn time, char length, and burn rate. The nonmetalized acid dyes used did influence certain flammability characteristics; however, the effects showed up in only 22% of the cases. Beneficial effects outnumbered adverse effects by a ratio of

2 to 1. Adverse effects occurred most frequently in ease of ignition. In addition, the 1,2,4-anthraquinone dyes most frequently affected flammability properties, while the azo dyes were less likely to change the flammability behavior of the fabric.

III. RESEARCH USING THE MUSHROOM APPAREL FLAMMABILITY TESTER

Since simulation of fire accidents is an important experimental approach to measuring the burn injury potential of fabrics and garments (39), a careful analysis of instruments used for such simulation is important. The MAFT is an instrument used for this purpose which has been under critical evaluation. Several research projects using the MAFT have been reported in the literature.

Pete (45) used the MAFT to evaluate cotton and cotton/polyester fabrics which had been treated with varying levels of the flame retardant Fyrol 76. Results showed higher levels of Fyrol were required for the fabric to pass the vertical test than the MAFT. In addition, the Fyrol add-on required to pass Class 1 of the MAFT was dependent on fabric weight, construction, and composition.

Thompson (55) compared the MAFT with the vertical test and the 45° angle test using cotton and cotton/polyester fabrics. The MAFT did not limit the use of fabrics as severely as the vertical test. Although all eight fabrics tested passed both the MAFT and the 45° angle test, the eight fabrics did not fall into the same classes as outlined in the Draft Proposed Standard (46); thus the MAFT would limit the end use for some of the fabrics, whereas the 45° angle test would not.

The same fabrics used in a study conducted by Galil and Lomartire (19) were burned on the MAFT by Krasny and Peacock (31) in an effort to evaluate a desirability function designed by Galil and Lomartire. Krasny and Peacock found that the MAFT classification and the desirability function gave the same results for only 5 out of 16 fabrics. Flame properties, fabric area destroyed, and weight loss used in the desirability function were not related to heat evolution from burning fabrics. Krasny and Peacock emphasized the importance of using heat transfer which relates directly to burn injury.

Galil and Lomartire (20) emphasized that they chose basic burning parameters related to quantity and rate since their interest was in measuring the degree of flammability of apparel fabrics rather than setting up a standard. Galil and Lomartire believe the MAFT is a pass/fail test which cannot rate fabrics according to their degree of flammability. They also criticized the MAFT's heat transfer measurements as being apparatus dependent.

Miller and Meiser (42) found a strong relationship between heat emission rate measured by convection calorimetry and MAFT heat transfer values. Miles (40) did not obtain such positive results in a comparison of the hazard potential measured by the Apparel Fire Modeling Apparatus (AFMA) and the MAFT classification. The AFMA includes a mobility feature which simulates the motion of an individual resulting in bodily contact with the burning fabric. Six of the 18 fabrics tested on the MAFT and AFMA showed inconsistencies. A lower AFMA hazard potential was obtained than the MAFT class indicated.

Concerns with the reliability, reproducibility, and stability of the MAFT led to research conducted by Segal and Drake (48,49) using two MAFTs at the Southern Regional Research Center. They found reproducible results can be obtained when the sensors and sensor signal processor are well constructed and the MAFT is properly calibrated against the lamps. Segal (48) also found the MAFT to be stable during a day's operation. In addition, the importance of testing specimens in both the warp and fill directions was indicated by the differences found in MAFT values according to specimen orientation during testing. Segal emphasized that the instrument can be used for research or developmental work if qualifications are made for it. The results obtained are relative, i.e., the results are not absolute in magnitude but indicate trends being detected by the one instrument being used.

IV. CELLULOSE DECOMPOSITION AND COMBUSTION

Current data indicate dyes may affect the course of decomposition and combustion of cellulose. Flammability testing by itself does not explain the mechanism by which cellulose pyrolytically degrades. Current knowledge of cellulose decomposition and combustion is the result of data on thermodynamics and kinetics of degradation provided by thermogravimetric analysis (TGA), differential thermal analysis (DTA), and differential scanning calorimetry (DSC) (22).

In general, the burning processes of textiles consist of heating, decomposition, combustion, and propagation. When sufficient heat is applied to a textile, decomposition begins and the textile discolors due to bond failure. As decomposition continues, tars and liquids are formed and these decompose further into combustible and non-combustible gases. The quantity of gases produced depends on the nature of the textile, environmental conditions, and the nature of added chemicals (47).

Combustion involves flaming, glowing, or smoldering, all of which are exothermic processes. Flaming takes place in the combustible vapors above the decomposing textile surface. Glowing occurs in an abundance of oxygen at the surface and in the gas phase just above the surface. Smolder is slow, suppressed exothermic combustion occurring beneath the surface in a limited oxygen supply. Smoke is evolved during smoldering. When the heat of combustion minus the heat loss to the surroundings is sufficient to bring the adjacent textile mass to the combustion temperature, propagation of the burning processes will be self-sustaining (47).

The first attempts at characterizing the thermal degradation of cellulose involved measuring the amounts of gases, chars, and tars produced. The production of large proportions of tars was found to favor high flammability (22,47). Levoglucosan is the main constituent of the tar-fraction obtained from cellulose pyrolysis. Levoglucosan is considered to be the major fuel which contributes to the flammability of cellulose (5).

The mechanism by which cellulose thermally degrades involves the depolymerization of cellulose to levoglucosan through scission of 1,4 glycosidic linkages followed by hydrogen abstraction and isomerization to glucosan (22). The mechanism is believed to be independent of the degree of crystallinity (47); however, the packing density of the cellulose chains does affect the course of the chemical processes which occur during decomposition. Decreased packing density causes a decrease in levoglucosan formation, while increased packing density hinders the side processes of hydrolysis and dehydration, thus increasing levoglucosan formation (22).

In relation to temperature, cellulose undergoes decomposition involving loss of water, producing a partially dehydrated species, at 220°C. Depolymerization and volatilization begin at about 280°C. The remaining reactions involve decomposition of the dehydrated cellulose through exothermic reactions (22). The temperature of the fabric substrate is maintained by the gas phase oxidation of cellulose decomposition products. If this gas phase oxidation is sufficiently vigorous to maintain a fabric substrate temperature which is high enough to effect pyrolytic degradation, propagation of flaming results. The rate of degradation must also be great enough to maintain an adequate fuel supply to the gas phase in order to sustain flame propagation (23). The flaming temperature of cotton is about 413°C and the glowing temperature about 600°C (47).

CHAPTER III

METHODOLOGY

Reported in this chapter are the experimental design and description of the fabrics, dyes, and sample preparation. The procedures for measurement of physical properties and flammability testing are reported along with statistical analysis of the data obtained in the study.

I. EXPERIMENTAL DESIGN

A 2 x 3 x 2 fully crossed and balanced design was used to test the effect of three independent variables (dye type, dye color, and dye level) on each of nine dependent variables (peak heat transfer rates, time to reach peak heat transfer rates, total heat transfer, total burn time, heat/time relationship, rate of flame spread, and heat of decomposition). In addition, undyed fabric was tested for comparison with the dyed fabric. The MAFT was used to measure the first seven dependent variables, the 45° angle tester was used to measure rate of flame spread, and heat of decomposition was measured on the DSC. The entire study was replicated twice.

II. FABRIC

A single weight of 100% plain woven cotton was obtained from TESTFABRICS, INC., Middlesex, New Jersey for use in this study. Physical properties of the fabric were determined as follows:

Fabric weight--Fabric weight was determined as specified in ASTM Method D1910-64 (3). Ten randomly selected 5.08 x 5.08 cm squares were weighed on a Mettler balance. The weight in grams was converted to ounces per square yard and grams per square meter.

Fabric thickness--ASTM Method D1777-64 (3) was used to determine fabric thickness. Ten randomly selected 5.08 x 5.08 cm squares were measured on an Electric Low Pressure Thickness Gauge to the nearest 0.001 of an inch.

Thread count--Thread count was determined in accordance with procedures specified in ASTM Method D1910-64 (3). The mean number of ends and picks of five randomly selected one inch areas were counted and recorded.

Cotton count--Fabric strips measuring 10 cm x 45 cm were cut from the warp and fill direction of the fabric and 10 yarns from each direction were removed. Yarn crimp was removed under a tension of 4 grams using a Shirley Crimp Tester. During crimp removal yarns were cut to a length of 40 cm. Yarns were weighed on a Mettler balance and denier was calculated according to the following formula:

$$\frac{W \times 9000}{L}$$

where W = the weight in grams of the specified length of yarn and L = the length of the yarn in meters. Denier was converted to cotton count (CC) as follows:

$$CC = \frac{5314.87}{\text{denier}}$$

Fabric cover--The thread count in the warp and filling directions was used to calculate cover (K) using the following formulae:

$$K_w = \frac{N_w}{\sqrt{CC_w}} \qquad K_f = \frac{N_f}{\sqrt{CC_f}}$$

$$K\ \text{fabric} = K_w + K_f$$

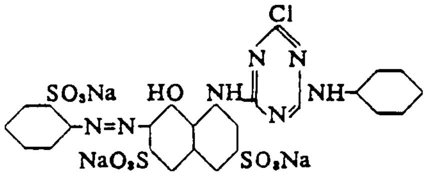
where N = number of threads designated either warp (w) or fill (f) and CC = cotton count, warp or fill.

III. DYES

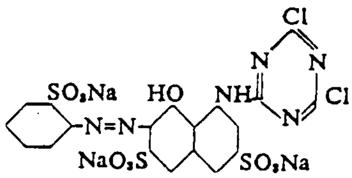
The following six dyes representing two types of reactive dyes (monochlorotriazine and dichlorotriazine) were used to dye the cotton fabric: CI Reactive Red 3 (Procion Red H-3B), CI Reactive Red 1 (Procion Red MX-2B), CI Reactive Yellow 2 (Procion Yellow H-5G), CI Reactive Yellow 1 (Procion Yellow MX-6G), CI Reactive Blue 5 (Procion Brilliant Blue H-GR), and CI Reactive Blue 4 (Procion Brilliant Blue MX-R). Chemical structures of the dyes are shown in Figure 1. Dyeing was done at Kimtex, Inc., Patterson, New Jersey. The undyed control fabric also was run through the dyeing process without the addition of dye.

IV. SAMPLE PREPARATION

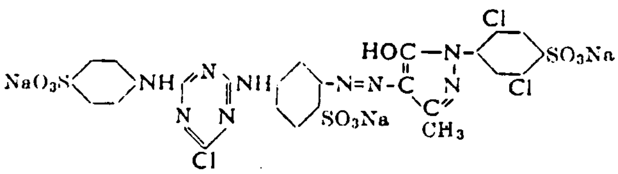
Fabrics were laundered once prior to cutting specimens for both physical tests and burning tests using AATCC Test Method 124-1975 (2). MAFT specimens were conditioned for at least 8 hours in a room with a temperature of 21 $\pm$ 1°C and relative humidity 65 $\pm$ 2%. The MAFT test instrument was located in a separate unconditioned room; therefore, each specimen was ignited within 60 seconds of its removal from the conditioned room.



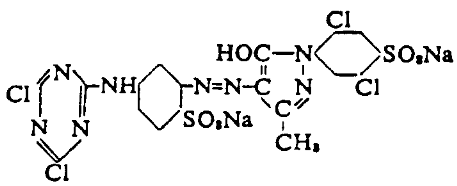
PROCION RED H-3B
(C.I. Reactive Red 3)



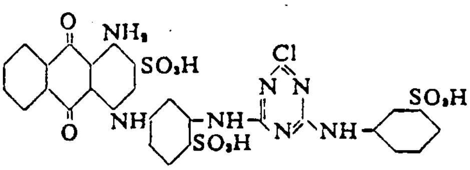
PROCION RED MX-2B
(C.I. Reactive Red 1)



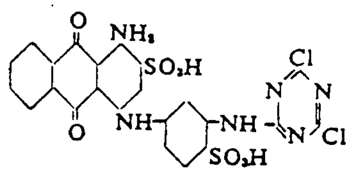
PROCION YELLOW H-5G
(C.I. Reactive Yellow 2)



PROCION YELLOW MX-6G
(C.I. Reactive Yellow 1)



PROCION BRILLIANT BLUE H-GR
(C.I. Reactive Blue 5)



PROCION BRILLIANT BLUE MX-R
(C.I. Reactive Blue 4)

Figure 1. Chemical Structures of Reactive Dyes.

V. FLAMMABILITY TESTING

MAFT

A sample consisted of four specimens randomly selected from each test fabric. Specimen dimensions were 32 cm x 61 cm, with the shorter dimension in the warp direction. Analysis of previous heat transfer test results on comparable cotton fabric revealed no difference between warp and fill specimens (55).

For heat transfer testing the MAFT was calibrated and cleaned according to specifications in the Draft Proposed Standard (46). Each specimen was mounted on the sensor using three metal clips spaced evenly around the top collar. Specimens were draped evenly around the sensor and the 2.5 cm overlap of the free edges pinned at the bottom with a straight pin. Each specimen was exposed to the ignition source for 3 seconds and allowed to burn to completion. The point at which burning ceased was recorded on the heat transfer curve which was automatically plotted on the recorder during the experiment. The sensor was cooled between each test to within 5°C of ambient conditions using a 23 cm electric fan.

Previous heat transfer testing on comparable cotton fabric indicated two main peaks are produced in the heat transfer curve (55). The heat transfer rate at the two peaks, the time to reach the heat transfer rate peaks, and total burn time were determined from each heat transfer curve. Total heat transfer was calculated by integration of the total area under each curve. Heat/time data were determined by integrating the area under each MAFT curve at 12 second intervals.

Average heat/time values for each of the samples were used to establish the heat/time relationship for each fabric.

45° Angle Tester

All fabrics were tested according to the specifications in AATCC Test Method 33-1962 (2). A sample consisted of 10 randomly selected specimens measuring 5.1 cm x 15.2 cm. Five specimens were cut from the warp direction and five specimens were cut from the filling direction. Each specimen was dried in an oven at 105 ± 3°C for 30 minutes, cooled in a desiccator, mounted, and exposed to the flame for 1 second. Burning times were recorded to the nearest 0.1 of a second.

DSC

A sample consisted of two randomly selected specimens from each test fabric. Aluminum sample pans were used for testing. The pan and lid were weighed together to the nearest 0.1 mg. Approximately 8 mg of fabric was added to each pan. The filled pans were weighed and the specimen weight determined by the difference.

DSC analyses were performed using a Perkin-Elmer DSC 2. Calibration was completed with a 6.4 mg tin reference standard. Each specimen was heated from 314°K to 773°K at a scan rate of 20°K/minute. The analyses were performed under a nitrogen atmosphere. Residue weights were determined following DSC analysis.

A linear baseline was drawn on each DSC curve according to the method outlined by Fredrick and Mentzer (18). The temperature at onset of degradation and the peak temperatures were determined from each curve. In addition, a ratio of peak heights was established for the dyed fabrics which had two peaks. Planimetry was used to graphically integrate the area bounded by the exotherm peak and the baseline. Heat of decomposition (in calories/gram) was calculated using the integrated area under the curve, the known ΔHf for the tin reference standard, and the Δmass of the specimen.

VI. STATISTICAL ANALYSIS

A one-way analysis of variance with orthogonal contrasts to test for dye main effects (dye type, dye level, and dye color), interactions, and dyed vs. undyed fabric was used for each of the dependent variables specified in the experimental design. The analysis was also used for the temperature at onset of degradation, the temperatures at the degradation peaks, and the peak heights ratio obtained in the DSC test. Further differences between undyed and dyed fabrics were determined using Waller-Duncan Multiple Range post-hoc tests. A significance level of 0.01 was used for the orthogonal contrasts and the Waller-Duncan test was used with a K-Ratio of 100.

Regression analysis was used to establish the heat/time relationship curves. The average heat/time values for each of the samples for each fabric were fit to the following exponential growth curve:

$$HE = A(1-e^{-Kt})^{\frac{1}{1-M}}$$

where HE represents the heat transferred at time t, A represents the total heat transferred by the fabric, K provides for rapid acceleration in the curve, and M provides for a slow initial rate of heat transfer.

The K and M parameters provide for curve characteristics which match the characteristics of the heat/time data for these fabrics. The Derksen injury curve (14), a curve which shows the time rate heat exposure which produces a second degree skin burn, was superimposed over the calculated heat/time relationship curves. The times at which the injury curve intersected the calculated heat/time relationship curve and intersected the average heat/time data values were determined. The A, K, and M values established by the regression analysis and the times to intersect the Derksen injury curve were also analyzed using the one-way analysis of variance with orthogonal contrasts and the Waller-Duncan post-hoc test.

CHAPTER IV

RESULTS AND DISCUSSION

Reported in this chapter are the results for the physical properties of weight, thickness, thread count, cotton count, and fabric cover for each test fabric. In addition, the results for the MAFT test, the 45° angle test, and the DSC test are reported for each test fabric.

Also reported is the effect of dye type, dye level, dye color, and dyed vs. undyed fabric on the peak heat transfer rates, time to reach peak heat transfer rates, total burn time, total heat transfer, heat/time relationship, rate of flame spread, temperature at onset of degradation, peak degradation temperatures, peak heights ratio, and heat of decomposition. A description of the results of the regression analysis used for the heat/time relationship is included as well as representative MAFT heat transfer curves and a description of the burning behavior of the test fabrics on the MAFT.

I. DYED FABRICS

Physical Properties

The mean results for the physical properties of weight, thickness, thread count, cotton count, and fabric cover are listed in Table I. The findings indicate that the cotton fabrics are equivalent in physical properties despite processing with different types, colors, and levels of reactive dyes.

Table I. Physical Properties of Fabrics.[a]

Fabric[b]	Weight		Thickness[d] (in Mills)	Thread Count[c] Warp / Fill	Cotton Count[e] Warp / Fill	Cover Factor[f]
	oz/yd²	g/m²				
MX Red 1%	3.97	135	15	130 / 80	44 / 45	31.46
MX Red 4%	3.93	133	15	129 / 80	45 / 47	30.89
H Red 1%	3.96	134	15	130 / 80	42 / 44	32.15
H Red 4%	3.95	134	15	130 / 80	43 / 43	31.79
MX Yellow 1%	3.85	131	15	130 / 81	45 / 44	31.47
MX Yellow 4%	3.82	129	15	130 / 81	43 / 44	31.92
H Yellow 1%	3.92	133	15	129 / 80	43 / 44	31.73
H Yellow 4%	3.74	127	15	130 / 80	45 / 43	31.58
MX Blue 1%	3.84	130	15	130 / 80	42 / 44	31.99
MX Blue 4%	3.91	132	15	130 / 80	41 / 44	32.36
H Blue 1%	3.85	130	15	130 / 80	44 / 43	31.70
H Blue 4%	3.89	132	15	130 / 80	43 / 44	31.89
Undyed	3.91	132	15	130 / 82	43 / 42	32.47

[a]All fabrics were purchased from Testfabrics, Inc., Middlesex, New Jersey and dyed at Kimtex, Inc., Patterson, New Jersey.

[b]Identified according to dye type, dye color, and dye level.

[c]Determined according to standard procedure ASTM D1910 (3).

[d]Determined according to standard procedure ASTM D1777 (3).

[e]5314.87/denier (the number of 840 yard hanks per pound of yarn).

[f]K(warp) + K(filling) = K(fabric cover).

MAFT Results

The mean results for the MAFT test are given in Table II. A one-way analysis of variance with orthogonal contrasts was used to test for differences in dye main effects (dye type, dye level, and dye color) and interactions on peak heat transfer rates, time to reach peak heat transfer rates, total burn time, and total heat transfer, as measured by the MAFT. Results of the six analyses are shown in Tables III-VIII.

The contrasts for three of the dependent variables--the heat transfer rate at the first peak, the time to reach the first heat transfer rate peak, and total burn time--were found to be non-significant. Significant differences were found in the heat transfer rate at the second peak, the time to reach the second heat transfer rate peak, and total heat transfer; however, not all the dye main effects and interactions were significant. The analysis results for each of these dependent variables will be discussed separately.

Heat transfer rate at the second peak. Dye level was the only significant effect for the heat transfer rate at the second peak (Table IV). Fabrics dyed at the 4% level exhibited significantly higher heat transfer rates at the second peak than fabrics dyed at the 1% level. The MX Red 4% dyed fabric was the only 4% fabric which had a heat transfer rate at the second peak in the same range as the 1% fabrics.

Time to reach the second heat transfer rate peak. The times to reach the second heat transfer rate peak were significantly different

Table II. Summary of MAFT Test Results.[a]

Fabric[b]	Heat Transfer Rate at 1st Peak ($J/cm^2/sec$)	Heat Transfer Rate at 2nd Peak ($J/cm^2/sec$)	Time to Reach 1st Heat Transfer Rate Peak (sec)	Time to Reach 2nd Heat Transfer Rate Peak (sec)	Burn Time (sec)	Total Heat Transfer (J/cm^2)
MX Red 1%	1.30	1.25	16.15	74.40	147.80	103.24
MX Red 4%	1.30	1.29	16.30	69.55	143.65	110.90
H Red 1%	1.32	1.41	16.65	77.05	142.90	105.50
H Red 4%	1.32	1.84	15.65	113.50	139.05	158.13
MX Yellow 1%	1.30	1.08	15.15	72.15	149.40	86.51
MX Yellow 4%	1.33	1.64	15.65	111.40	133.90	147.97
H Yellow 1%	1.26	1.21	15.40	68.15	145.90	101.26
H Yellow 4%	1.39	1.90	16.90	115.65	139.90	175.84
MX Blue 1%	1.31	1.31	16.30	65.40	144.15	98.24
MX Blue 4%	1.28	1.91	15.90	118.00	149.50	186.27
H Blue 1%	1.26	1.23	16.30	88.75	141.90	111.44
H Blue 4%	1.31	1.87	16.65	114.15	140.50	167.25
Undyed	1.28	1.00	16.40	82.25	158.75	90.37

[a]Mean values for all variables; N=8.

[b]Identified according to dye type, dye color, and dye level.

Table III. Analysis of Variance for Heat Transfer Rate at First Peak.

Source	df	Sum of Squares	Mean Square	F
Model	12	0.0293	0.0024	0.73
Contrast				
Type	1	0.0002	0.0002	0.06
Level	1	0.0063	0.0063	1.90
Color	2	0.0034	0.0017	0.51
Type x Level	1	0.0051	0.0051	1.53
Type x Color	2	0.0011	0.0006	0.17
Level x Color	2	0.0084	0.0042	1.27
Type x Level x Color	2	0.0036	0.0018	0.53
Dyed vs. Undyed	1	0.0011	0.0011	0.34
Error	13	0.0432	0.0033	
Total	25	0.0725		

*Significant at the 0.01 level.

Table IV. Analysis of Variance for Heat Transfer Rate at Second Peak.

Source	df	Sum of Squares	Mean Square	F
Model	12	2.62	0.22	8.62*
Contrast				
Type	1	0.16	0.16	6.32
Level	1	1.46	1.46	57.61*
Color	2	0.09	0.04	1.71
Type x Level	1	0.05	0.05	1.99
Type x Color	2	0.18	0.09	3.49
Level x Color	2	0.20	0.10	4.03
Type x Level X Color	2	0.04	0.02	0.17
Dyed vs. Undyed	1	0.45	0.45	17.69*
Error	13	0.33	0.03	
Total	25	2.95		

*Significant at the 0.01 level.

Table V. Analysis of Variance for Time to Reach First Heat Transfer Rate Peak.

Source	df	Sum of Squares	Mean Square	F
Model	12	6.59	0.55	1.57
Contrast				
Type	1	0.74	0.74	2.10
Level	1	0.20	0.20	0.58
Color	2	1.18	0.59	1.69
Type x Level	1	0.06	0.06	0.17
Type x Color	2	0.68	0.34	0.97
Level x Color	2	2.16	1.08	3.09
Type x Level x Color	2	1.38	0.69	1.97
Dyed vs. Undyed	1	0.19	0.19	0.53
Error	13	4.55	0.35	
Total	25	11.14		

*Significant at the 0.01 level.

Table VI. Analysis of Variance for Time to Reach Second Heat Transfer Rate Peak.

Source	df	Sum of Squares	Mean Square	F
Model	12	10654.88	887.91	15.18*
Contrast				
Type	1	773.72	773.72	12.54*
Level	1	6425.55	6425.55	109.86*
Color	2	686.91	343.46	5.87
Type x Level	1	83.25	83.25	1.42
Type x Color	2	542.22	271.11	4.64
Level x Color	2	878.51	439.25	7.51*
Type x Level x Color	2	1173.54	586.77	10.03*
Dyed vs. Undyed	1	131.17	131.17	2.24
Error	13	760.34	58.49	
Total	25	11415.22		

*Significant at the 0.01 level.

Table VII. Analysis of Variance for Total Burn Time.

Source	df	Sum of Squares	Mean Square	F
Model	12	908.40	75.70	1.37
Contrast				
Type	1	55.51	55.51	1.01
Level	1	108.80	108.80	1.97
Color	2	12.30	6.15	0.11
Type x Level	1	1.55	1.55	0.03
Type x Color	2	56.02	28.01	0.51
Level x Color	2	162.13	81.06	1.47
Type x Level x Color	2	66.40	33.20	0.60
Dyed vs. Undyed	1	445.69	445.69	8.08
Error	13	717.16	55.17	
Total	25	1625.56		

*Significant at the 0.01 level.

Table VIII. Analysis of Variance for Total Heat Transfer.

Source	df	Sum of Squares	Mean Square	F
Model	12	29843.75	2486.98	21.12*
Contrast				
Type	1	1240.99	1240.99	10.54*
Level	1	19287.07	19287.07	163.82*
Color	2	1851.37	925.69	7.86*
Type x Level	1	111.63	111.63	0.95
Type x Color	2	909.08	454.54	3.86
Level x Color	2	2129.80	1064.90	9.05*
Type x Level x Color	2	1504.72	752.36	6.39
Dyed vs. Undyed	1	2809.08	2809.08	23.86*
Error	13	1530.50	117.73	
Total	25	31374.25		

*Significant at the 0.01 level.

for the dyed fabrics due to dye type, dye level, the two-way interaction of level and color, and the three-way interaction of type, level, and color (Table VI). Fabrics dyed with the Procion H dyes took a longer time to reach the second heat transfer rate peak than the Procion MX dyed fabrics. The time to reach the second heat transfer rate peak was also longer for fabrics dyed at the 4% level than those dyed at the 1% level.

Because of the significant level x color interaction, the means were graphed to provide an understanding of differences (Figure 2). As with the heat transfer rate at the second peak, the MX Red 4% dyed fabric exhibited a time to reach the second heat transfer rate peak which was lower than the other 4% fabrics. Thus, the difference between the levels of dye did not remain constant across the levels of the color factor for this variable.

Graphs of means for the type x level x color interaction are shown in Figure 3. The time to reach the second heat transfer rate peak at the 1% level was greatest for H Blue and MX Red fabrics and at the 4% level was greatest for H Yellow and MX Blue fabrics. Again, the MX Red 4% dyed fabric greatly contributed to the significant interaction.

Total heat transfer. All dye main effects (dye type, dye level, and dye color) and the interaction level x color were significant for total heat transfer (Table VII). Total heat transfer was higher for the Procion H dyed fabrics, blue fabrics, and fabrics dyed at the 4% level. The red fabrics as a group had the lowest total heat

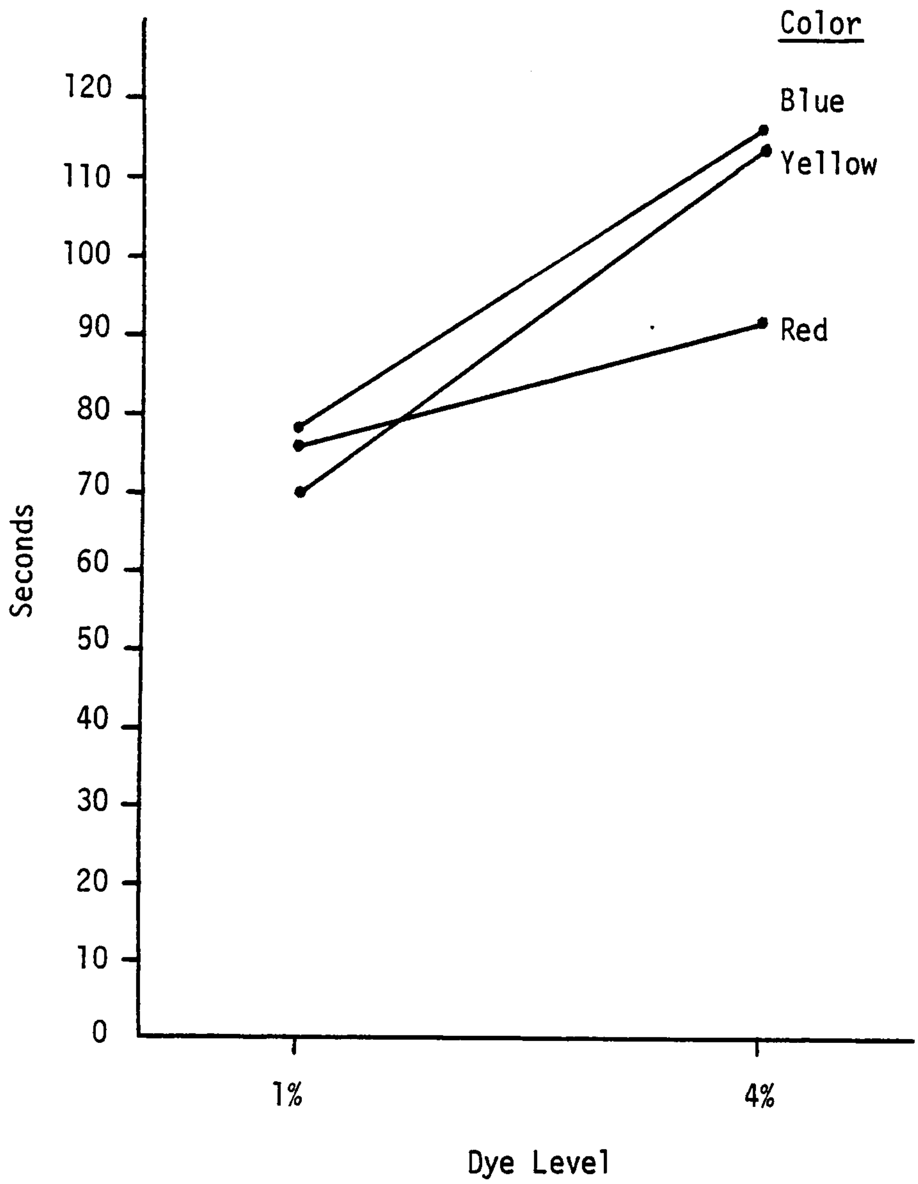


Figure 2. Mean Values of Time to Reach the Second Heat Transfer Rate Peak for Dye Level and Dye Color.

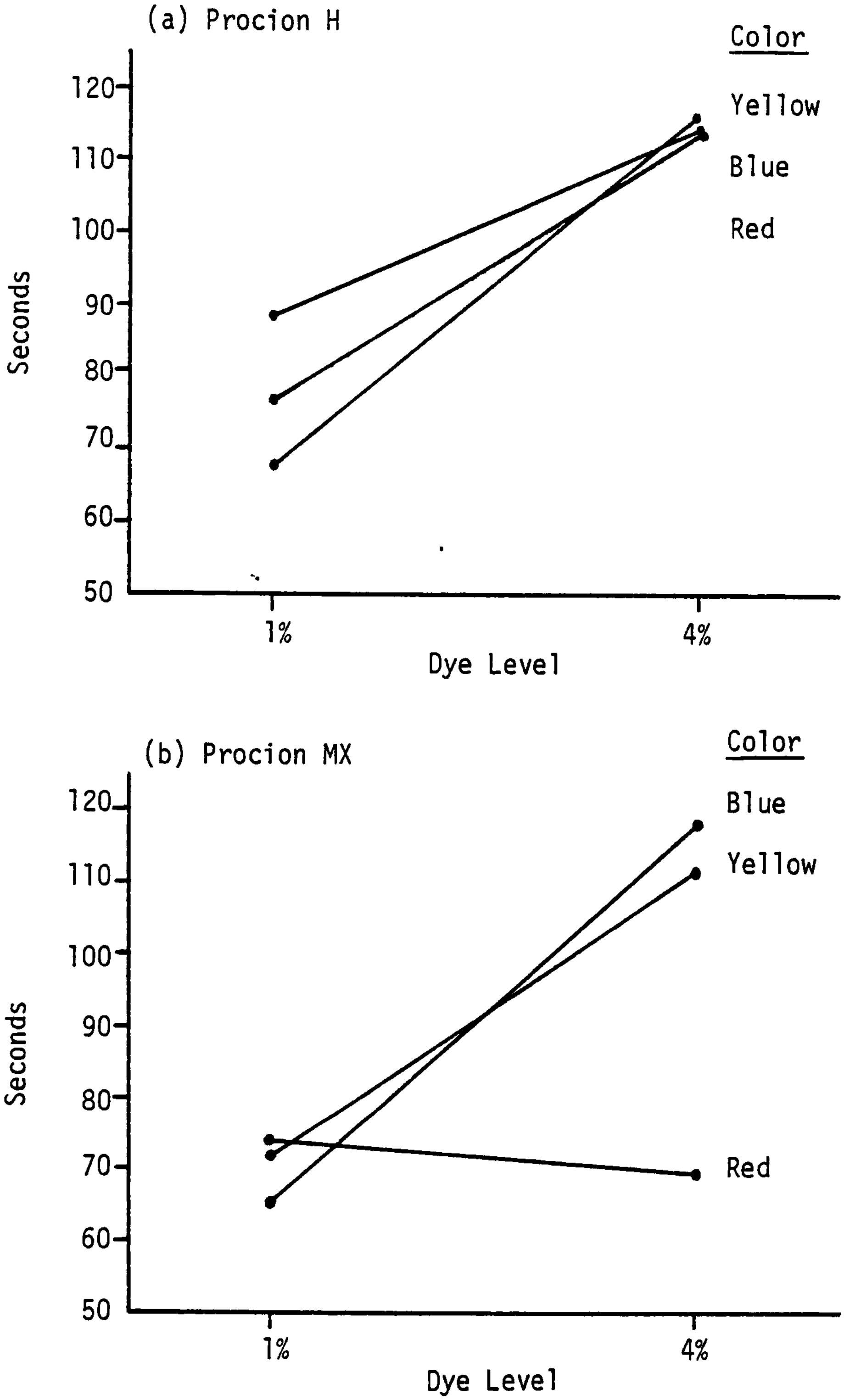


Figure 3. Mean Values of Time to Reach the Second Heat Transfer Rate Peak for Dye Type, Dye Level, and Dye Color.

transfer due to the MX Red 4% dyed fabric which had a total heat transfer in the same range as the 1% fabrics rather than the higher value characteristic of the other 4% fabrics.

The MX Red 4% fabric also contributed to the significant interaction level x color for total heat transfer. The graph of means in Figure 4 shows the significant interaction level x color. The Red 4% fabrics show a smaller increase in total heat transfer across the levels of dye than the blue and yellow fabrics.

<u>Heat/time relationship</u>. Heat/time data were obtained by integration of each of the MAFT heat transfer curves at 12 second intervals. The characteristics of the integrated MAFT heat transfer curves for the fabrics tested were a slow initial rate of heat transfer followed by a rapid acceleration in the rate and finally a leveling off as the total amount of heat transferred by the fabric was reached. Therefore, to examine the heat/time relationship obtained in the integrated curves, the average heat transfer values for each sample at 12-second intervals were fit to an exponential growth curve of the form

$HE = A(1-e^{-Kt})^{\frac{1}{1-M}}$. In this equation HE represents the heat transferred at time t, A represents the total heat transferred by the fabric, K provides for the rapid acceleration in the curve, and the M provides for the slow initial rate of heat transfer.

The effect of changes in the values of the parameters A, K, and M on the curve was examined. Results of the analyses are shown by the curves in the Appendix, Figures 28-30. The curves indicate that a fabric with a high K value and a low M value transfers heat

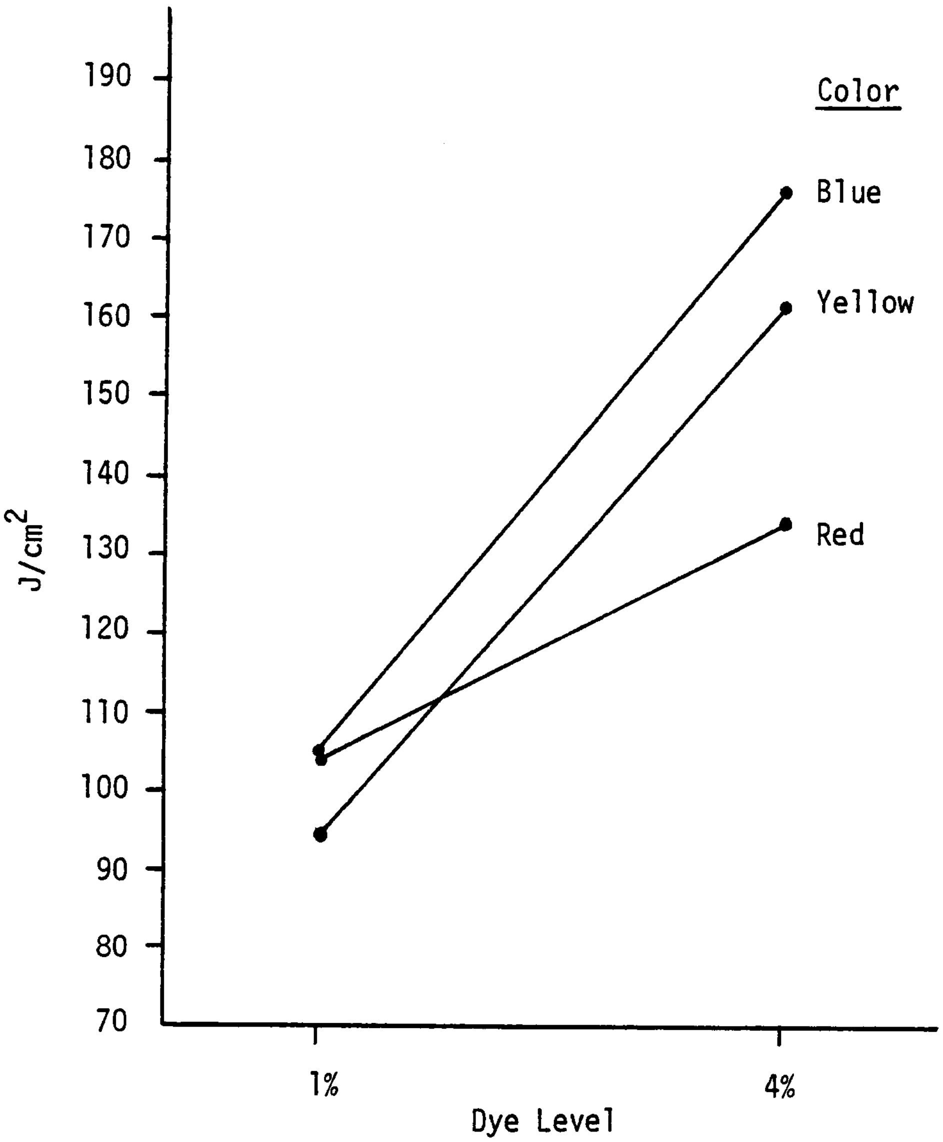


Figure 4. Mean Values of Total Heat Transfer for Dye Level and Dye Color.

rapidly and quickly reaches its total heat transfer while a fabric with a low K value and high M value transfers heat more slowly. The mean parameter values obtained for the fabrics tested are listed in Table IX along with R^2 values for the regression curves.

The one-way analysis of variance procedure with orthogonal contrasts was used to test for differences in dye main effects (dye type, dye level, and dye color) and interactions on the A, K, and M values. Results of the three analyses are shown in Tables X-XII.

Dye level significantly affected all three parameter values. Fabrics dyed at the 4% level exhibited higher A values, lower K values, and higher M values than fabrics dyed at the 1% level. Results indicate the 4% fabrics transferred heat more slowly than the 1% fabrics but ultimately transferred more heat.

Dye level was the only significant effect for parameters K and M. Parameter A, like its close counterpart total heat transfer, showed significant differences due to dye type, dye color, and the interactions level x color and type x level x color as well as dye level. As with total heat transfer, the A values were higher for the Procion H dyed fabrics, blue fabrics, and fabrics dyed at the 4% level. The red fabrics as a group had the lowest A value due to the MX Red 4% dyed fabric which had an A value in the same range as the 1% fabrics rather than the higher value characteristic of the other 4% fabrics.

The graph of means in Figure 5 shows the significant interaction level x color for parameter A. Again, the MX Red 4% fabric contributed to the significant interaction. Figure 6 shows the significant interaction type x level x color for parameter A. The value of

Table IX. Heat/Time Relationship Curve Values.

Fabric[a]	$HE = A(1-e^{-Kt})^{\frac{1}{1-M}}$			R^2	t_{cal}[b]	t_{data}[c]
	A	K	M			
MX Red 1%	110.12	0.0235	0.6445	0.9911	27	21
MX Red 4%	115.95	0.0240	0.6735	0.9943	27	21
H Red 1%	110.81	0.0260	0.6855	0.9925	27	21
H Red 4%	167.89	0.0210	0.7405	0.9894	37	21
MX Yellow 1%	92.85	0.0250	0.6245	0.9918	25	21
MX Yellow 4%	156.41	0.0220	0.7215	0.9906	34	21
H Yellow 1%	106.50	0.0240	0.6280	0.9949	26	21
H Yellow 4%	184.97	0.0200	0.7525	0.9901	38	22
MX Blue 1%	103.42	0.0260	0.6440	0.9932	25	21
MX Blue 4%	196.42	0.0195	0.7725	0.9901	40	23
H Blue 1%	119.54	0.0215	0.6550	0.9910	28	22
H Blue 4%	176.81	0.0200	0.7370	0.9914	37	22
Undyed	99.30	0.0205	0.5690	0.9914	26	21

[a]Identified according to dye type, dye color, and dye level.

[b]Time of intersection for the Derksen injury curve and the calculated heat/time relationship curve.

[c]Time of intersection for the Derksen injury curve and the average heat/time data.

Table X. Analysis of Variance for Parameter A.

Source	df	Sum of Squares	Mean Square	F
Model	12	32417.58	2701.47	21.77*
Contrast				
Type	1	1390.65	1390.65	11.21*
Level	1	21028.43	21028.43	169.50*
Color	2	2121.34	1060.67	8.55*
Type x Level	1	154.28	154.28	1.24
Type x Color	2	890.70	445.35	3.59
Level x Color	2	2326.45	1163.23	9.38*
Type x Level x Color	2	1908.14	954.07	7.69*
Dyed vs. Undyed	1	2597.60	2597.60	20.94*
Error	13	1612.82	124.06	
Total	25	34030.40		

*Significant at the 0.01 level.

Table XI. Analysis of Variance for Parameter K.

Source	df	Sum of Squares	Mean Square	F
Model	12	0.0001305	0.0000109	3.72*
Contrast				
Type	1	0.0000094	0.0000094	3.21
Level	1	0.0000634	0.0000634	21.68*
Color	2	0.0000141	0.0000071	2.41
Type x Level	1	0.0000004	0.0000004	0.13
Type x Color	2	0.0000033	0.0000017	0.56
Level x Color	2	0.0000033	0.0000017	0.56
Type x Level x Color	2	0.0000278	0.0000139	4.75
Dyed vs. Undyed	1	0.0000090	0.0000090	3.08
Error	13	0.0000380	0.0000029	
Total	25	0.0001685		

*Significant at the 0.01 level.

Table XII. Analysis of Variance for Parameter M.

Source	df	Sum of Squares	Mean Square	F
Model	12	0.08760	0.00730	7.01*
Contrast				
Type	1	0.00232	0.00232	2.23
Level	1	0.04438	0.04438	42.60*
Color	2	0.00187	0.00094	0.90
Type x Level	1	0.00001	0.00001	0.01
Type x Color	2	0.00441	0.00221	2.12
Level x Color	2	0.00584	0.00292	2.80
Type x Level x Color	2	0.00179	0.00090	0.86
Dyed vs. Undyed	1	0.02700	0.02700	25.91*
Error	13	0.01354	0.00104	
Total	25	0.10114		

*Significant at the 0.01 level.

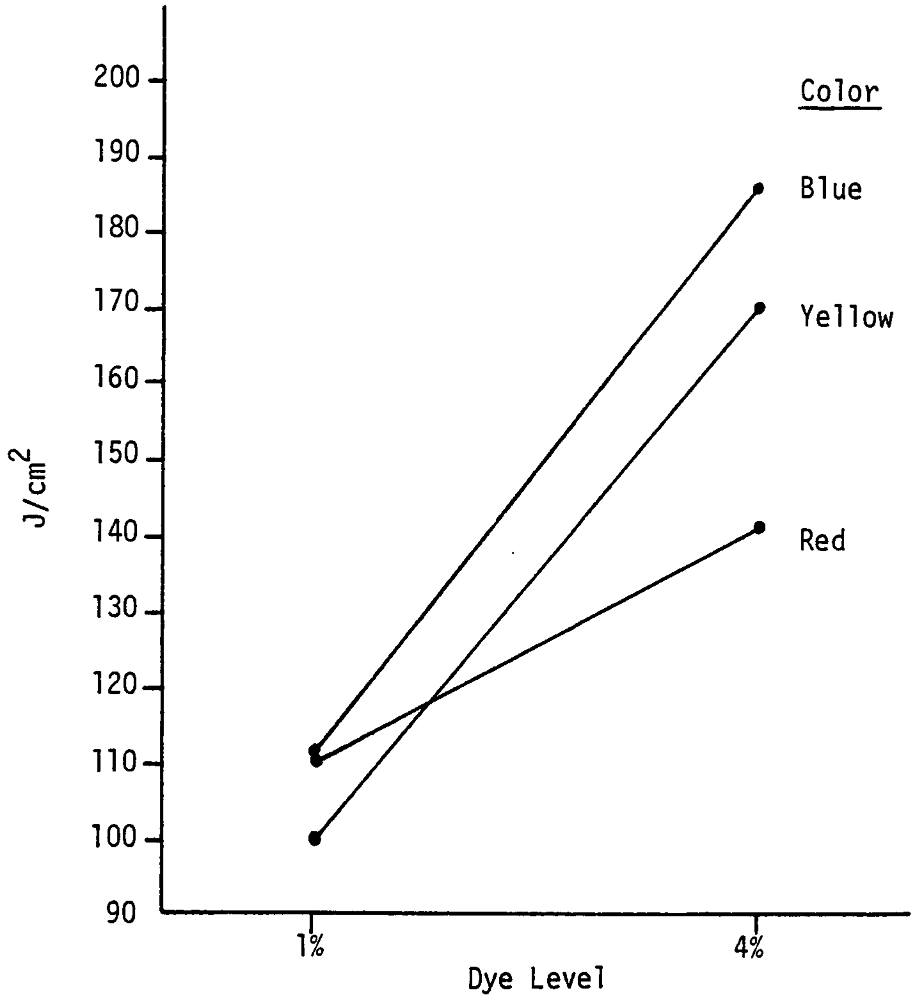


Figure 5. Mean Values of Parameter A for Dye Level and Dye Color.

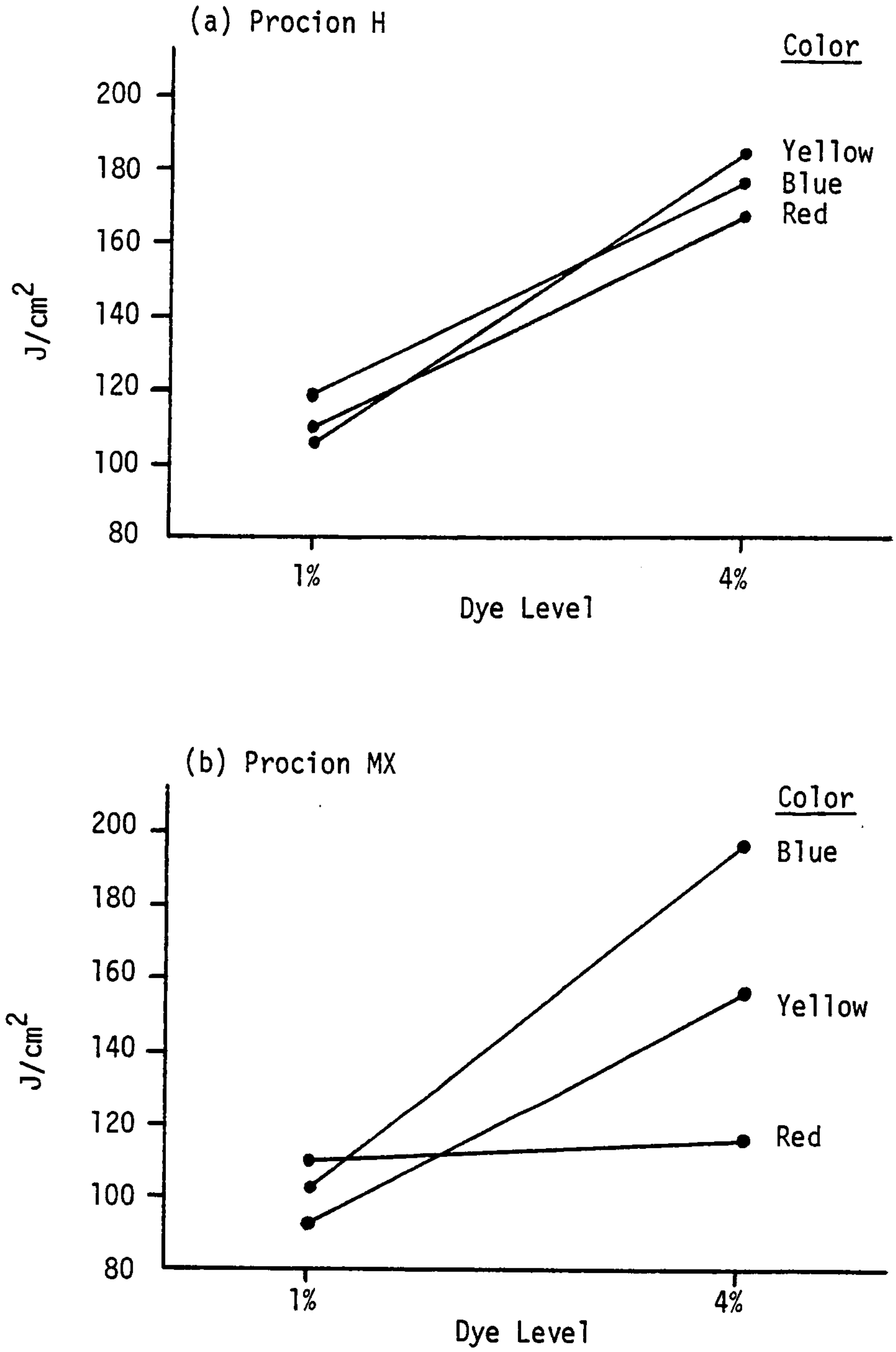


Figure 6. Mean Values of Parameter A for Dye Type, Dye Level, and Dye Color.

parameter A at the 1% level was greatest for H Blue and MX Red fabrics and at the 4% level was greatest for H Yellow and MX Blue fabrics.

Figures 7-12 illustrate the heat-time relationship curves obtained using this regression analysis on the average heat/time data for each fabric. The Derksen injury curve (14), a curve which shows the time rate heat exposure which produces a second degree skin burn, was superimposed over the calculated heat/time relationship curves. The time at which the curves intersected was noted as an indication of the time available for wearer reaction before severe burn injury would occur. The intersection times for both the calculated curve and the average heat/time data values are listed in Table IX. The intersection times reveal that the early portion of these heat/time curves is the most important in relation to flammability hazard and wearer reaction time.

A comparison of the calculated and data intersection times as well as a visual analysis of the heat/time curves in Figures 7-12 reveals that the rate of curve acceleration for the 4% fabrics in the 20 to 50 second area is slower in the calculated curve than the data suggests. The calculated curves do not appear to fit the data for the 4% fabrics between 20 and 50 seconds. Since the derivative for a curve established using the equation

$$HE = A(1-e^{-Kt})^{\frac{1}{1-M}}$$

is a curve with one peak, this regression analysis forces the data to conform to a one-peaked curve. This analysis provides a better fit for the 1% fabrics which do not have a large, distinct second peak.

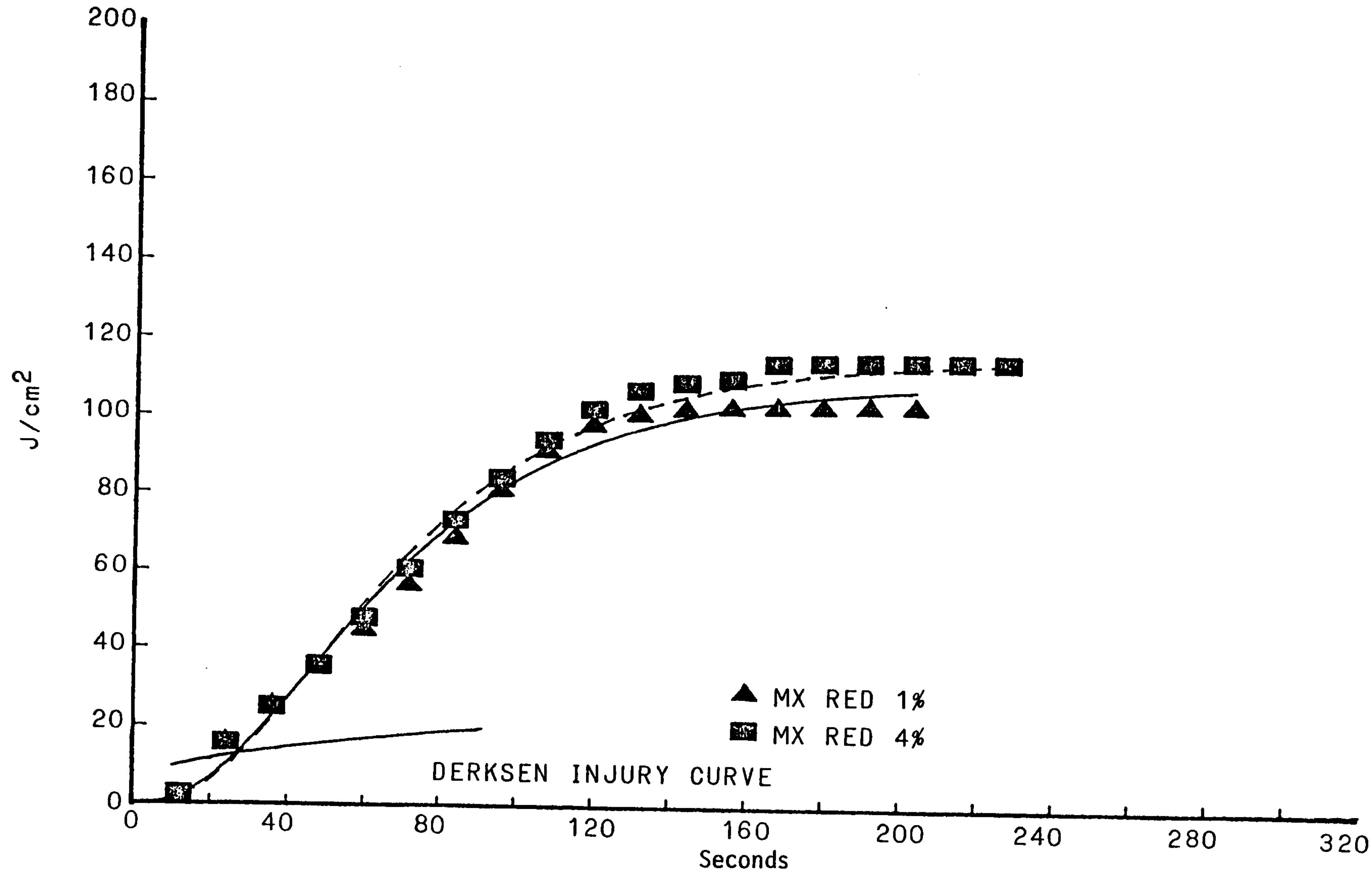


Figure 7. Heat/Time Relationship for MX Red Fabrics.

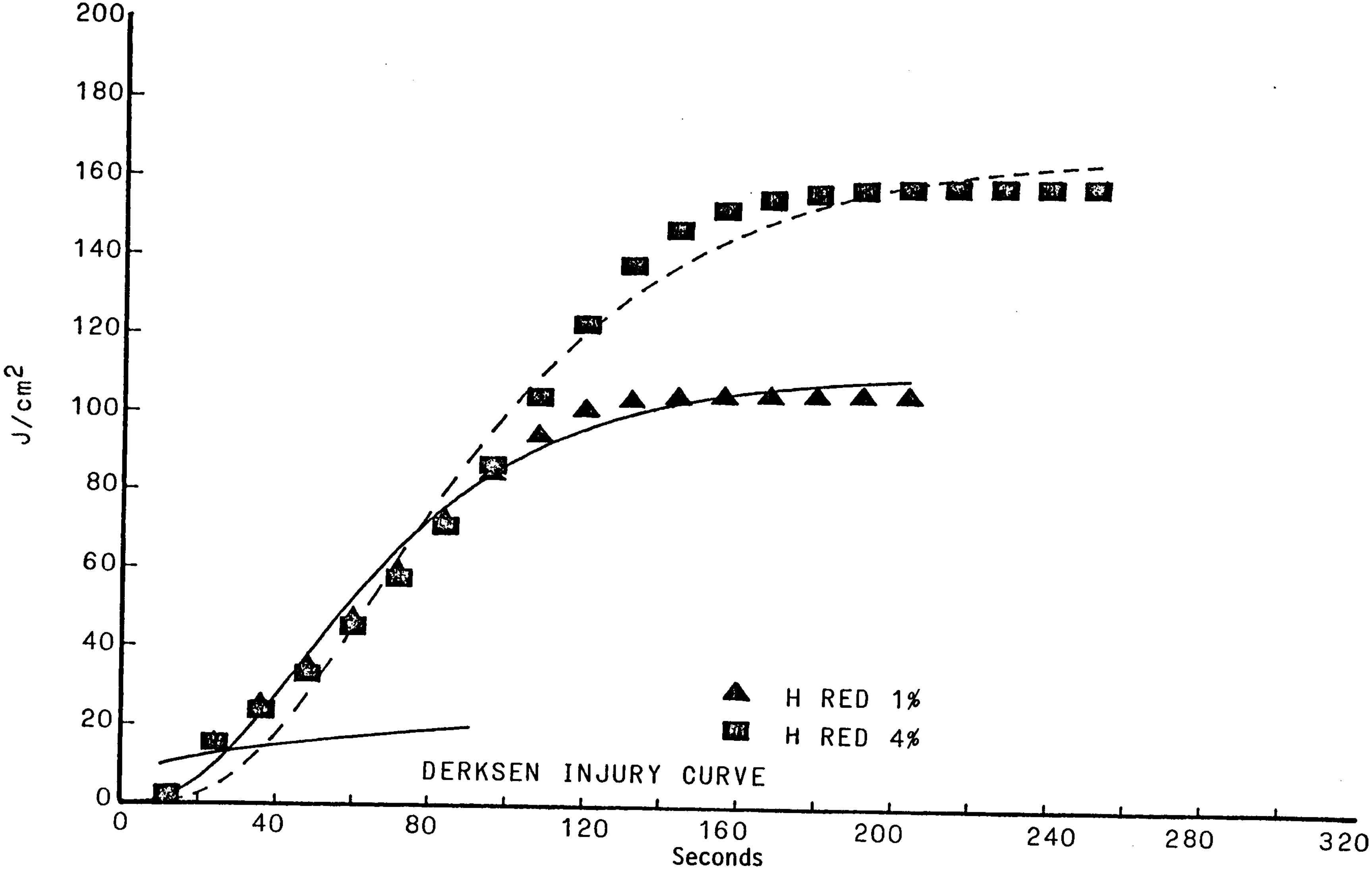


Figure 8. Heat/Time Relationship for H Red Fabrics.

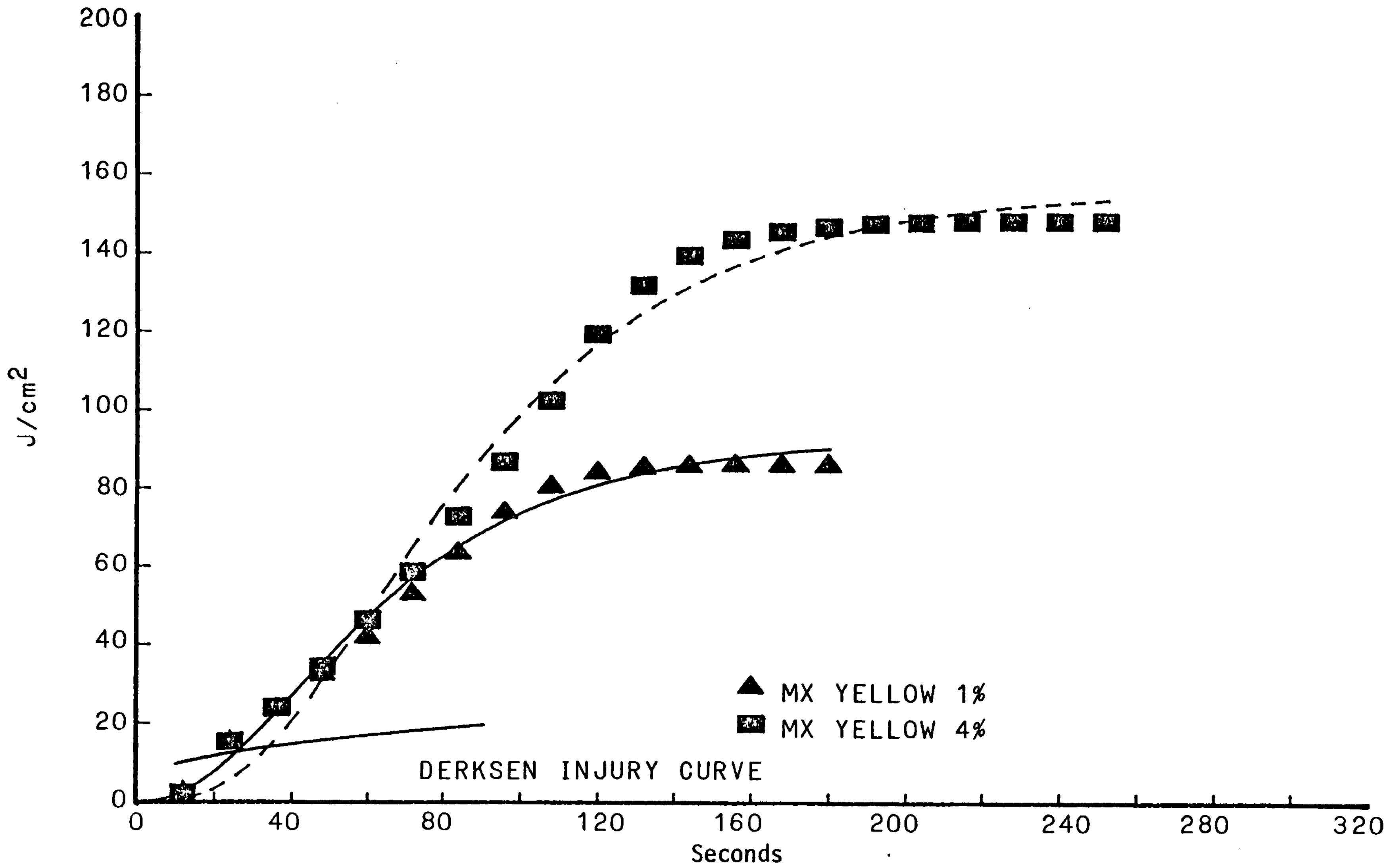


Figure 9. Heat/Time Relationship for MX Yellow Fabrics.

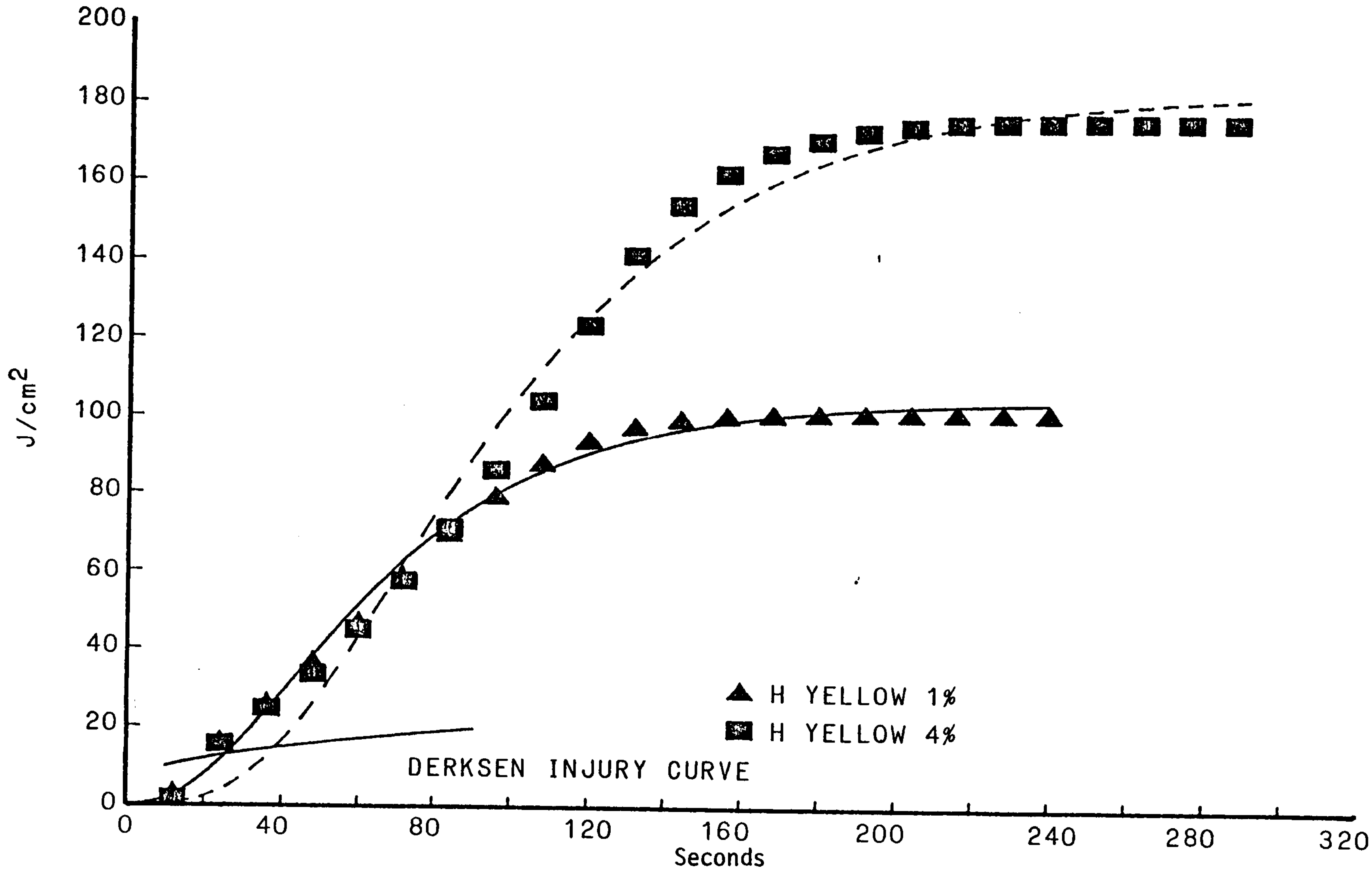


Figure 10. Heat/Time Relationship for H Yellow Fabrics.

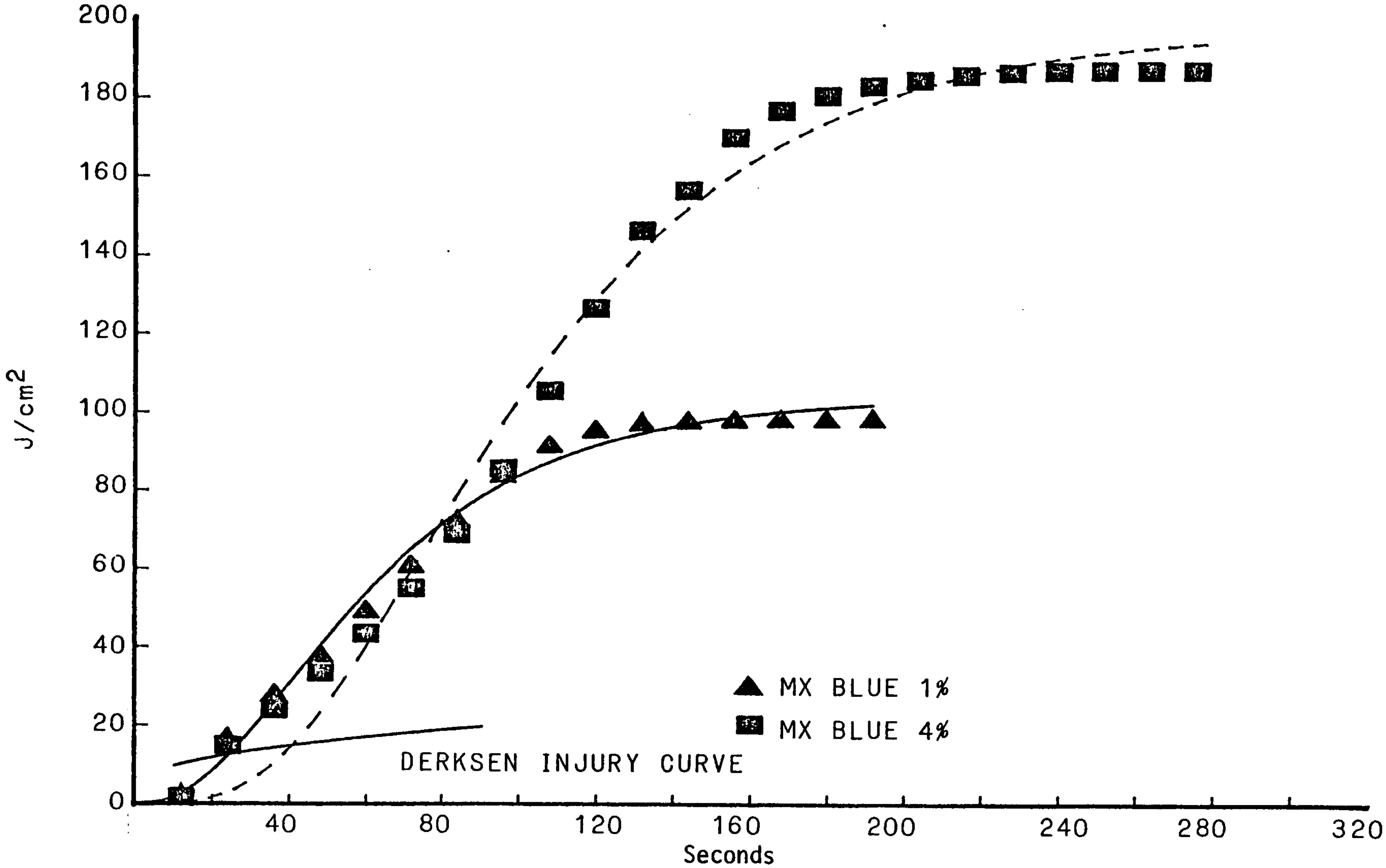


Figure 11. Heat/Time Relationship for MX Blue Fabrics.

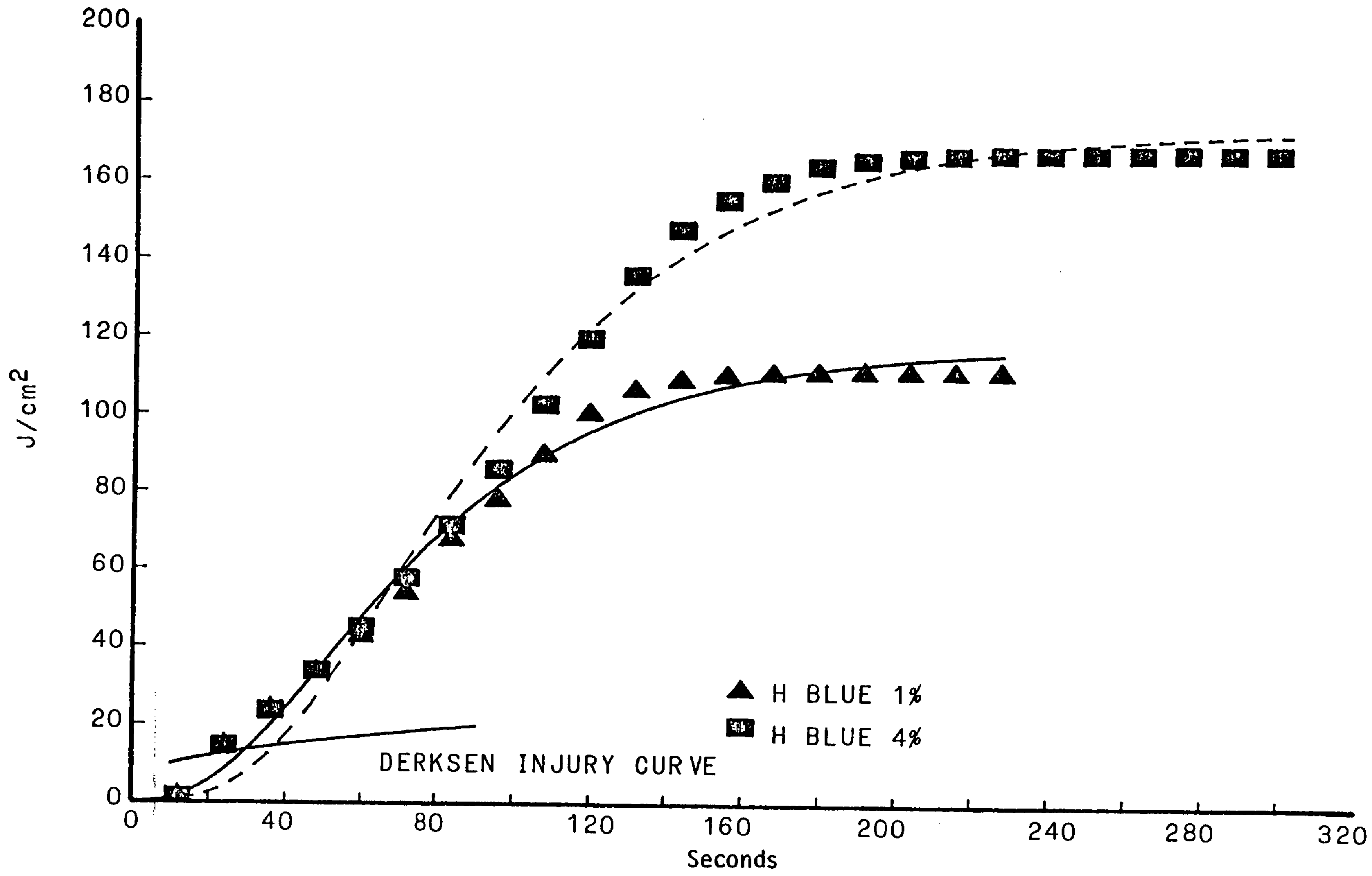


Figure 12. Heat/Time Relationship for H Blue Fabrics.

The one-way analysis of variance procedure with orthogonal contrasts was used to test for differences in dye main effects (dye type, dye level, and dye color) and interactions on the times to intersect the Derksen injury curve for both the calculated heat/time curves and the average heat/time data values for each sample. Results are shown in Tables XIII and XIV.

There is no difference in the heat/time data intersection times for the 12 dyed fabrics tested; however, the calculated curve intersection times yielded a significant difference due to dye level. This finding indicates, as suggested, that the initial rate of curve acceleration for the 4% fabrics is slower than the rate the data indicates.

Although the M values and Derksen injury curve intersection times for the calculated heat/time curves indicate that the 4% fabrics, with the exception of MX Red 4%, retard heat transfer during the first 50 to 80 seconds of burning, the rate and time of the retardation of initial heat transfer is not significant in the actual data. Consequently, the data indicate no difference in the hazard potential of the dyed fabrics during the first minute of burning.

Summary of MAFT results. In summary, the 12 dyed fabrics exhibited similar values for the heat transfer rate at the first peak, the time to reach the first heat transfer rate peak, and total burn time. All fabrics dyed at the 4% level, with the exception of the MX Red 4% fabric, exhibited higher values than fabrics dyed at the 1% level for the heat transfer rate at the second peak, the time to reach the second heat transfer rate peak, total heat transfer, and

Table XIII. Analysis of Variance for t_{cal}.[a]

Source	df	Sum of Squares	Mean Square	F
Model	12	795.85	66.32	6.71*
Contrast				
Type	1	35.04	35.04	3.55
Level	1	513.38	513.38	51.94*
Color	2	40.08	20.04	2.03
Type x Level	1	7.04	7.04	0.71
Type x Color	2	28.08	14.04	1.42
Level x Color	2	61.75	30.88	3.12
Type x Level x Color	2	68.08	34.04	3.44
Dyed vs. Undyed	1	42.39	42.39	4.29
Error	13	128.50	9.88	
Total	25	924.35		

[a]Time of intersection for the Derksen injury curve and the calculated heat/time relationship curve.

*Significant at the 0.01 level.

Table XIV. Analysis of Variance for t_{data}.[a]

Source	df	Sum of Squares	Mean Square	F
Model	12	24.15	2.01	3.27
Contrast				
Type	1	0.38	0.38	0.61
Level	1	5.04	5.04	8.19
Color	2	3.25	1.63	2.64
Type x Level	1	0.38	0.38	0.61
Type x Color	2	3.25	1.63	2.64
Level x Color	2	6.58	3.29	5.35
Type x Level x Color	2	5.25	2.68	4.27
Dyed vs. Undyed	1	0.03	0.03	0.05
Error	13	8.00	0.62	
Total	25	32.15		

[a]Time of intersection for the Derksen injury curve and the average heat/time data.

*Significant at the 0.01 level.

parameters A and M. Parameter K values were lower for the 4% dyed fabrics.

In terms of heat/time relationship the regression analysis results indicated the 4% dyed fabrics transfer heat more slowly initially, but ultimately transfer a greater amount of heat than the 1% fabrics. However, the heat/time data and Derksen injury curve intersection times revealed that there was no significant difference in the initial heat transfer and hazard potential of the 12 dyed fabrics.

The MX Red 4% fabric exhibited values for all significant MAFT variables which were closer to the 1% values than the other 4% values. Visual examination and reflectance readings were completed to evaluate the comparative amounts of dye in the fabrics, but this evaluation did not explain the differences in the MX Red 4% fabric's behavior. The MX Red 4% fabric contributed to the significant level x color interaction for time to reach the second heat transfer rate peak, total heat transfer and parameter A.

The Procion H fabrics exhibited higher values than the Procion MX fabrics for time to reach the second heat transfer rate peak, total heat transfer, and parameter A. The blue fabrics as a group had higher values than the yellow and red fabrics for total heat transfer and parameter A. Overall, the MX Blue 4% fabric had the highest values for all of the significant MAFT variables except parameter K. MX Blue 4% had the lowest value for parameter K.

45° Angle Test Results

The mean results for the 45° angle test are listed in Table XV. A one-way analysis of variance with orthogonal contrasts was used to test for differences in dye main effects (dye type, dye level, and dye color) and interactions on the rate of flame spread. Results of the analysis are shown in Table XVI.

Dye level was the only significant effect for the rate of flame spread. The 4% fabrics had longer flame spread times than the 1% fabrics; however, none of the differences between 1% and 4% fabrics were more than two seconds.

These results appear to be consistent with the MAFT results. MAFT heat/time data indicated that the 4% fabrics transferred slightly less heat than the 1% fabrics during the first 12 seconds of burning. However, the Derksen injury curve showed the critical time for these fabrics to between 20 and 40 seconds. At 20 seconds of burning, there is no difference between the 12 dyed fabrics in relation to flammability hazard.

DSC Results

The mean results for the DSC are given in Table XVII. A one-way analysis of variance with orthogonal contrasts was used to test for differences in dye main effects (dye type, dye level, and dye color) and interactions on the temperature at onset of degradation, the degradation temperature at the first peak, the degradation temperature at the second peak, the peak heights ratio, and the heat of decomposition. Results of the five analyses are shown in Tables XVIII-XXII. Each of these dependent variables will be discussed separately.

Table XV. Summary of 45° Angle Test Results.

Fabric[a]	Flame Spread[b] (sec)
MX Red 1%	11.2
MX Red 4%	11.8
H Red 1%	11.6
H Red 4%	12.1
MX Yellow 1%	10.6
MX Yellow 4%	11.4
H Yellow 1%	11.2
H Yellow 4%	12.6
MX Blue 1%	10.6
MX Blue 4%	12.1
H Blue 1%	11.1
H Blue 4%	12.2
Undyed	11.7

[a]Identified according to dye type, dye color, and dye level.

[b]Mean values; N=20.

Table XVI. Analysis of Variance for Rate of Flame Spread.

Source	df	Sum of Squares	Mean Square	F
Model	12	9.236	0.770	4.14*
<u>Contrast</u>				
Type	1	1.550	1.550	8.35
Level	1	6.100	6.100	32.84*
Color	2	0.198	0.099	0.53
Type x Level	1	0.004	0.004	0.02
Type x Color	2	0.476	0.238	1.28
Level x Color	2	0.586	0.293	1.58
Type x Level x Color	2	0.258	0.129	0.69
Dyed vs. Undyed	1	0.065	0.065	0.35
Error	13	2.415	0.186	
Total	25	11.651		

*Significant at the 0.01 level.

Table XVII. Summary of DSC Test Results.[a]

Fabric[b]	Temperature at Onset of Degradation (°K)	Temperature at 1st Degradation Peak (°K)	Temperature at 2nd Degradation Peak (°K)	Ratio of Peak Heights (h_1/h_2)	Heat of Decomposition (cal/g)
MX Red 1%	579	641	659	1.58	156.12
MX Red 4%	572	633	652	1.33	162.61
H Red 1%	572	633	651	1.50	166.55
H Red 4%	569	627	649	1.37	128.50
MX Yellow 1%	578	639	657	1.53	154.07
MX Yellow 4%	571	635	653	1.36	156.17
H Yellow 1%	579	642	659	1.52	154.07
H Yellow 4%	571	636	653	1.31	152.16
MX Blue 1%	552	641	658	1.50	138.32
MX Blue 4%	549	636	657	1.21	123.84
H Blue 1%	576	644	659	1.67	127.39
H Blue 4%	559	632	652	1.32	164.70
Undyed	588	649	-	-	120.52

[a]Mean values for all variables; N=2.

[b]Identified according to dye type, dye color, and dye level.

Table XVIII. Analysis of Variance for Temperature at Onset of Degradation.

Source	df	Sum of Squares	Mean Square	F
Model	12	2987.62	248.97	30.68*
Contrast				
Type	1	108.38	108.38	13.35*
Level	1	330.04	330.04	40.67*
Color	2	1220.58	610.29	75.20*
Type x Level	1	18.38	18.38	2.26
Type x Color	2	520.75	260.38	32.08*
Level x Color	2	20.58	10.29	1.27
Type x Level x Color	2	87.75	43.88	5.41
Dyed vs. Undyed	1	681.16	681.16	83.93*
Error	13	105.50	8.12	
Total	25	3093.12		

*Significant at the 0.01 level.

Table XIX. Analysis of Variance for Degradation Temperature at First Peak.

Source	df	Sum of Squares	Mean Square	F
Model	12	847.38	70.62	10.14*
Contrast				
Type	1	30.38	30.38	4.36
Level	1	273.38	273.38	39.27*
Color	2	117.25	58.63	8.42*
Type x Level	1	7.04	7.04	1.01
Type x Color	2	90.25	45.13	6.48
Level x Color	2	14.25	7.13	1.02
Type x Level x Color	2	20.58	10.29	1.48
Dyed vs. Undyed	1	294.26	294.26	42.27*
Error	13	90.50	6.96	
Total	25	937.88		

*Significant at the 0.01 level.

Table XX. Analysis of Variance for Degradation Temperature at Second Peak.

Source	df	Sum of Squares	Mean Square	F
Model	11	296.33	26.94	6.34*
Contrast				
Type	1	32.67	32.67	7.69
Level	1	130.67	130.67	30.75*
Color	2	62.58	31.29	7.36*
Type x Level	1	2.67	2.67	0.63
Type x Color	2	39.08	19.54	4.60
Level x Color	2	1.08	0.54	0.13
Type x Level x Color	2	27.58	13.79	3.25
Error	12	51.00	4.25	
Total	23	347.33		

*Significant at the 0.01 level.

Table XXI. Analysis of Variance for Peak Heights Ratio.

Source	df	Sum of Squares	Mean Square	F
Model	11	0.40375	0.03670	25.99*
Contrast				
Type	1	0.00570	0.00570	4.04
Level	1	0.32900	0.32900	232.92*
Color	2	0.00223	0.00111	0.79
Type x Level	1	0.00004	0.00004	0.03
Type x Color	2	0.03423	0.01712	12.12*
Level x Color	2	0.02343	0.01172	8.29*
Type x Level x Color	2	0.00910	0.00455	3.22
Error	12	0.01695	0.00141	
Total	23	0.42070		

*Significant at the 0.01 level.

Table XXII. Analysis of Variance for Heat of Decomposition.

Source	df	Sum of Squares	Mean Square	F
Model	12	6541.29	545.11	15.97*
Contrast				
Type	1	0.83	0.83	0.02
Level	1	12.16	12.16	0.36
Color	2	1237.05	618.53	18.12*
Type x Level	1	1.75	1.75	0.05
Type x Color	2	735.62	367.81	10.78*
Level x Color	2	746.64	373.32	10.94*
Type x Level x Color	2	2340.06	1170.03	34.28*
Dyed vs. Undyed	1	1467.18	1467.18	42.99*
Error	13	443.70	34.13	
Total	25	6984.99		

*Significant at the 0.01 level.

Temperature at onset of degradation. All dye main effects and the interaction type x color were significant for the temperature at onset of degradation (Table XVIII). The temperature was highest for Procion H fabrics, the 1% fabrics, and yellow fabrics. The red fabrics had values similar to the yellow fabrics while the blue fabrics as a group had the lowest temperature at the onset of degradation.

The differences between the levels of dye color did not remain constant across the levels of dye type. Figure 13 illustrates the type x color interaction with a graph of means. Yellow and blue fabrics exhibited a decrease in the temperature at onset of degradation from Procion H to Procion MX with the blue fabrics showing a greater rate of decrease. The red fabrics yielded an increase in temperature at onset of degradation from Procion H to Procion MX.

The low temperature values for the Procion MX Blue fabrics, evident in Figure 13, contributed to the dye type significant effect for this variable. Dye type was not significant for the peak degradation temperatures while both dye level and dye color were significant.

Degradation temperature at the first peak. Both dye level and dye color were significant for the degradation temperature at the first peak (Table XIX). Dye type and all interactions were found to be non-significant. The degradation temperature was highest for the 1% fabrics and the blue fabrics. The yellow fabrics had values similar to the blue fabrics while the red fabrics as a group had the lowest degradation temperature at the first peak.

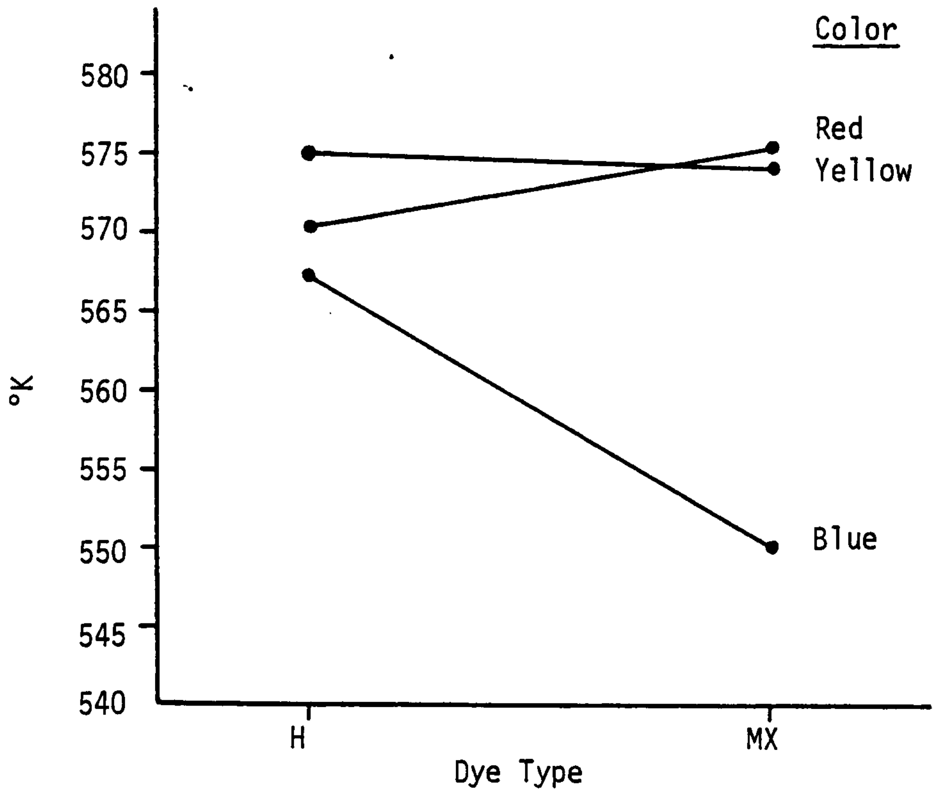


Figure 13. Mean Values of Temperature at Onset of Degradation for Dye Type and Dye Color.

Degradation temperature at the second peak. The analysis for the degradation temperature at the second peak gave results similar to those for the first peak. Both dye level and dye color were found to be significant (Table XX). The 1% fabrics and the blue fabrics had the highest degradation temperatures at the second peak. Values for the yellow fabrics were similar to the blue fabrics, but the red fabrics as a group had a lower degradation temperature at the second peak.

Peak heights ratio. In order to compare the relative size of the two degradation peaks a ratio of peak heights (h_1/h_2) was calculated. The peak heights ratio for the 12 dyed fabrics exhibited differences due to dye level and the interactions type x color and level x color. The 1% dyed fabrics had a higher peak heights ratio than the 4% fabrics, with the values indicating that the two peaks for the 4% fabrics were closer to being the same height.

Figures 14 and 15 show graphs of means for the type x color and level x color interactions. The low peak heights ratio exhibited by the MX Blue 4% fabric contributed to both interactions. The red and yellow fabrics yielded a slight increase in the peak heights ratio from Procion H to Procion MX; however, the blue fabrics yielded a sharp decrease in the ratio from Procion H to Procion MX (Figure 14). All colors yielded a decrease in the peak heights ratio from the 1% to the 4% level with the blue fabrics exhibiting the greatest decrease (Figure 15). The graphs reveal that the peak heights ratio is highest for H Blue and 1% Blue fabrics and lowest for MX Blue and 4% Blue fabrics.

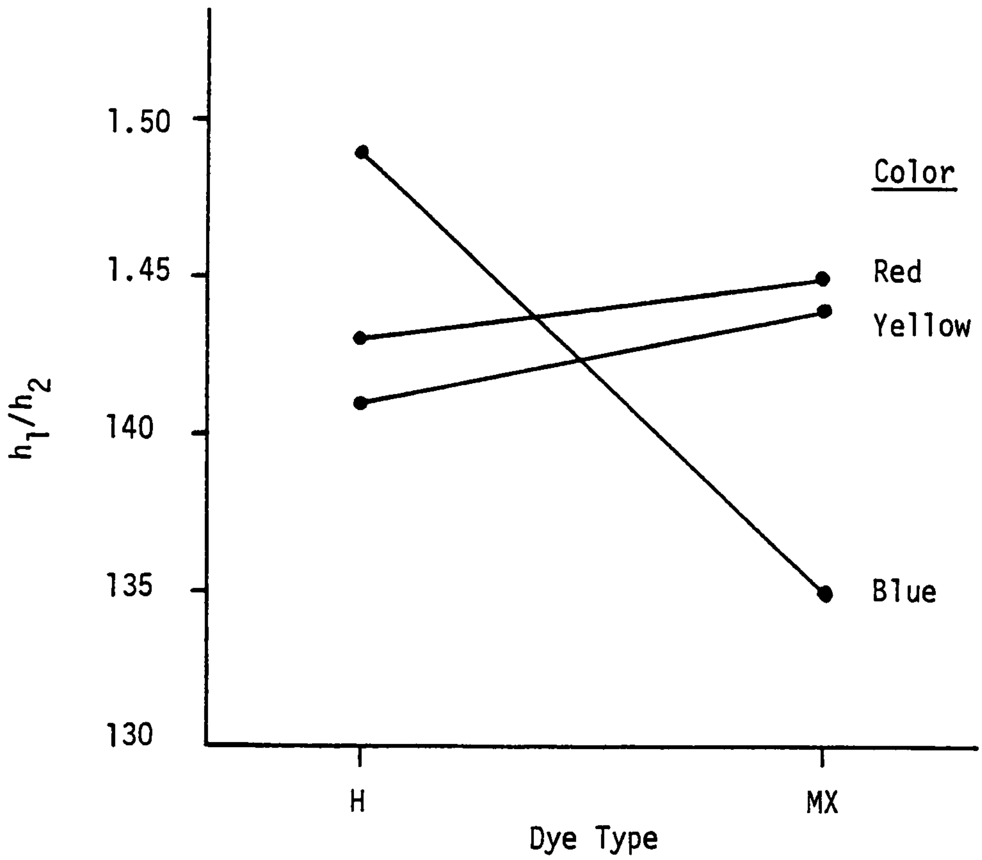


Figure 14. Mean Values of Peak Heights Ratio for Dye Type and Dye Color.

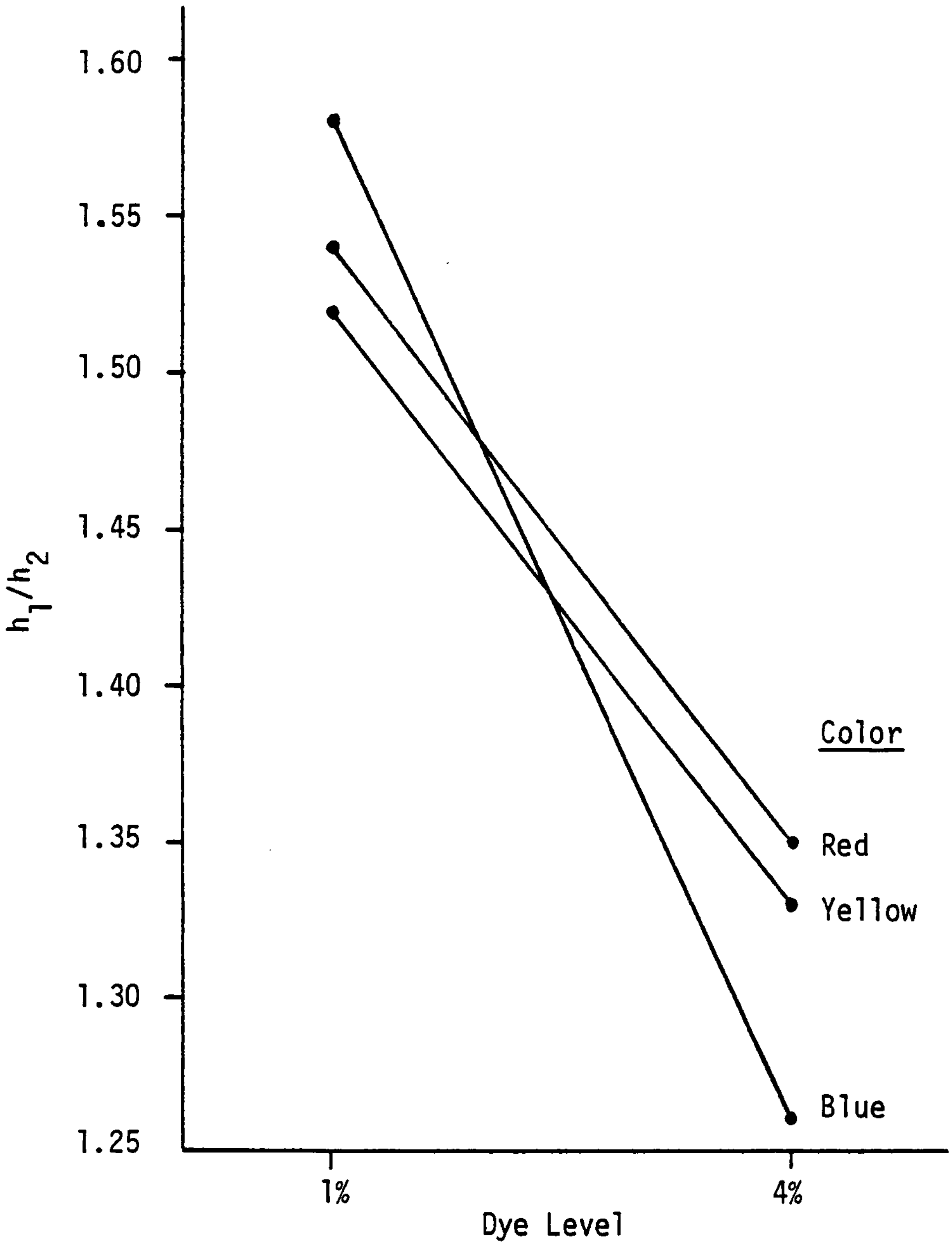


Figure 15. Mean Values of Peak Heights Ratio for Dye Level and Dye Color.

Heat of decomposition. Dye color and the interactions type x color, level x color, and type x level x color were found to be significant for heat of decomposition. The significant interactions create difficulty in examining the dye color main effect alone. Graphs of means for the interactions are illustrated in Figures 16-18.

The type x color interaction represented in Figure 16 reveals that the yellow fabrics have the highest heat of decomposition among Procion H fabrics and the red fabrics have the highest heat of decomposition among Procion MX fabrics. The yellow fabrics yielded a slight increase and the red fabrics a greater increase in heat of decomposition from Procion H to Procion MX. Heat of decomposition decreased sharply for the blue fabrics from Procion H to Procion MX.

Heat of decomposition differences between levels of color also did not remain constant across the dye levels. Figure 17 illustrates the level x color interaction. Heat of decomposition values for yellow fabrics were similar between the dye levels. Red fabrics exhibited a decrease in heat of decomposition and blue fabrics an increase from the 1% to the 4% level. The red fabrics have the highest value for this variable at the 1% level and the yellow fabrics have the highest value at the 4% level.

The dissimilarity of the two graphs in Figure 18 illustrates the type x level x color interaction. The Procion H Red and Blue fabrics exhibited the widest differences in heat of decomposition. For both dye types and at both dye levels the yellow fabrics show the greatest consistency. The Procion H Red 1%, H Blue 4%, MX Red 1%, and MX Red 4% had the highest heats of decomposition while the Procion H Blue 1%,

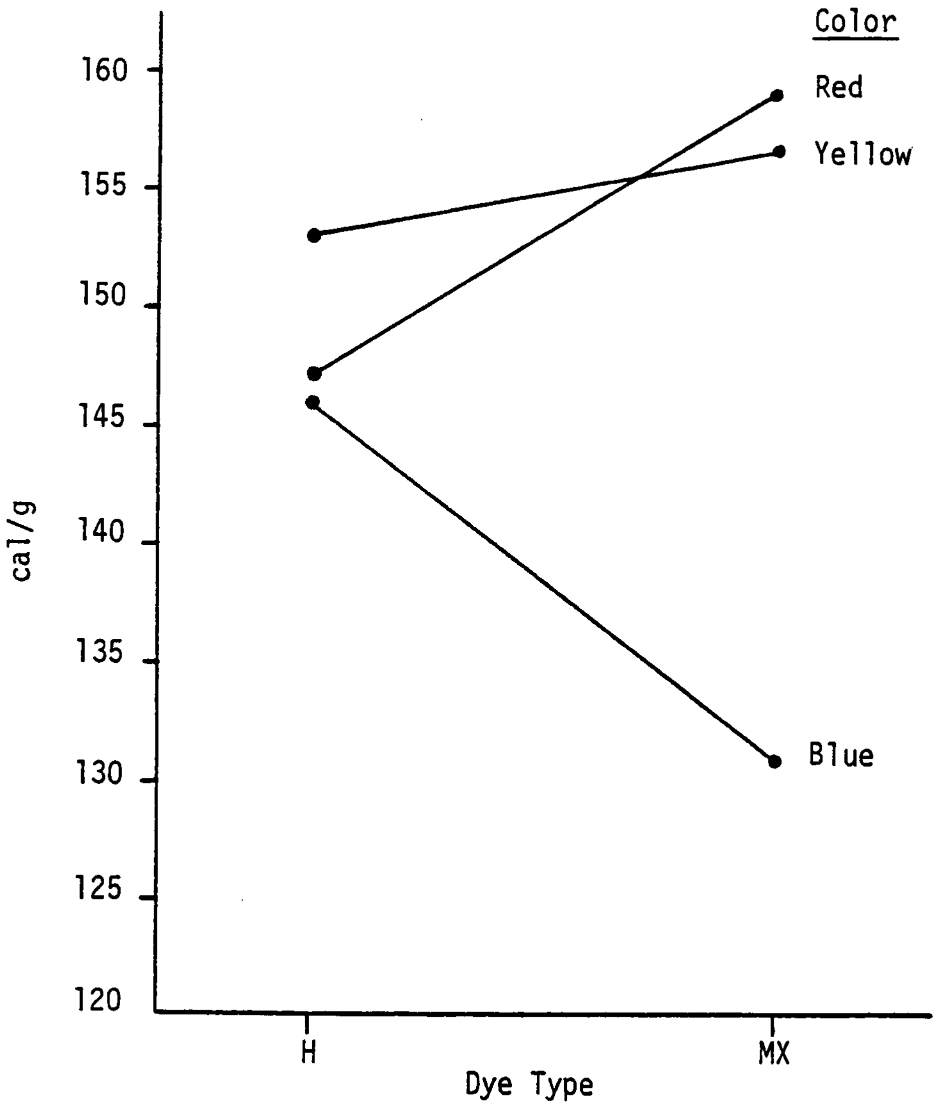


Figure 16. Mean Values of Heat of Decomposition for Dye Type and Dye Color.

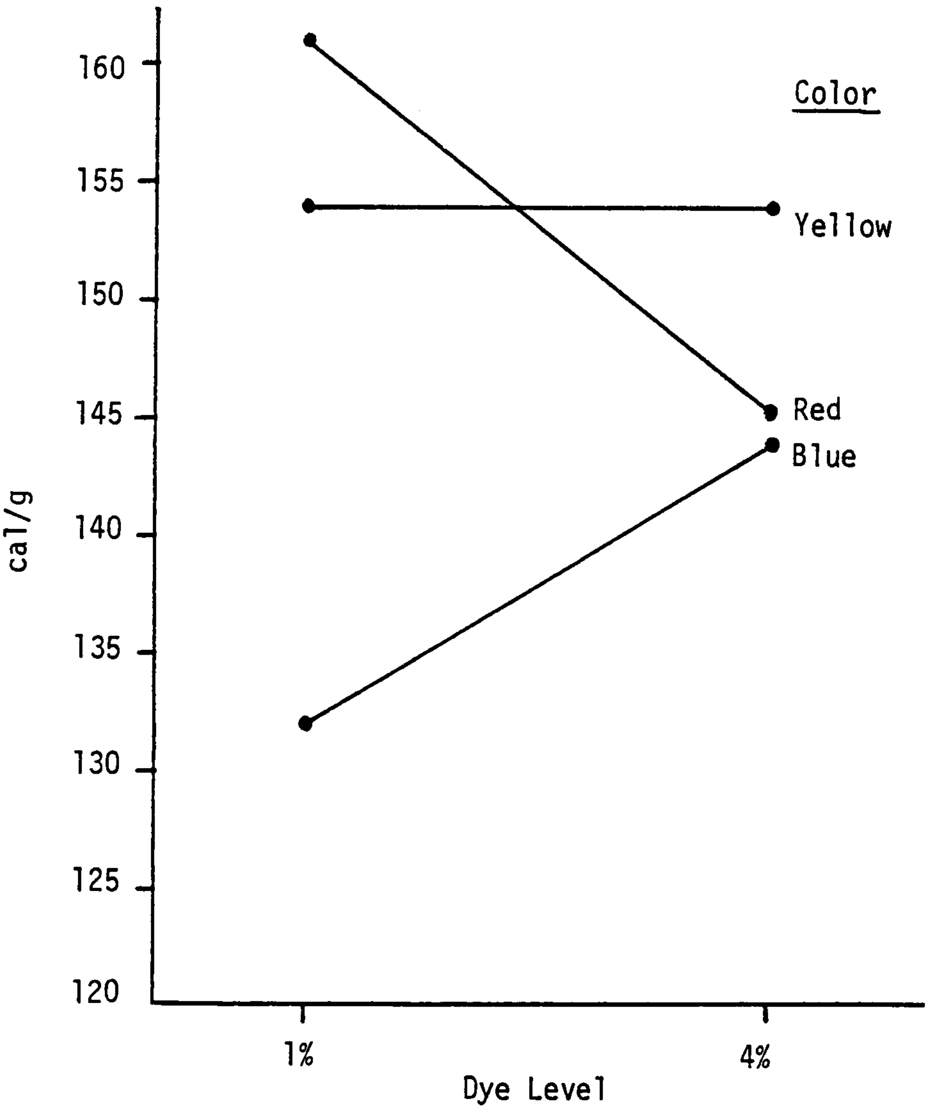


Figure 17. Mean Values of Heat of Decomposition for Dye Level and Dye Color

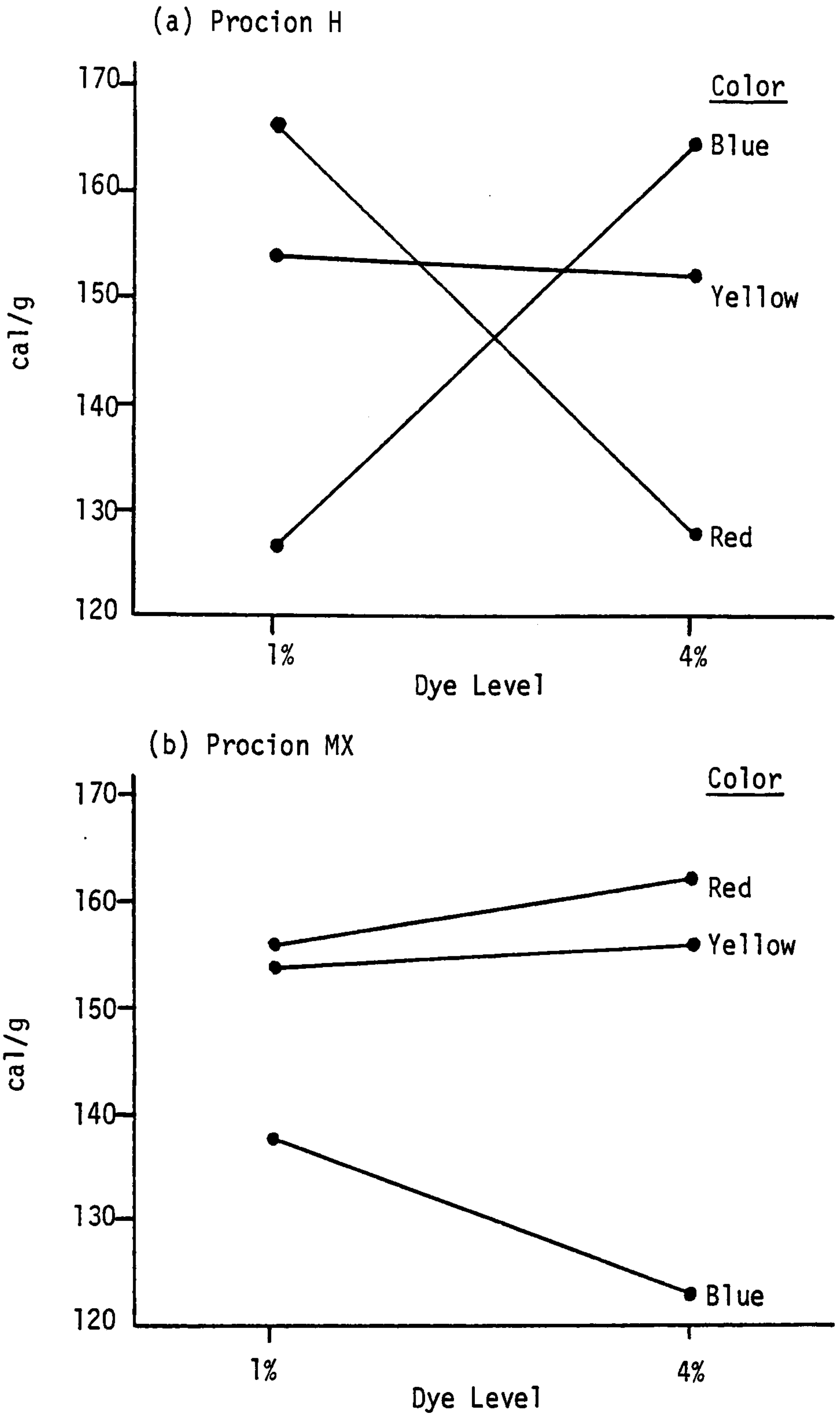


Figure 18. Mean Values of Heat of Decomposition for Dye Type, Dye Level, and Dye Color.

H Red 4%, MX Blue 1%, and MX Blue 4% had the lowest heats of decomposition.

Summary of DSC results. The DSC testing yielded differences for all of the dependent variables with the majority of differences related to dye level and dye color. The 4% fabrics exhibited significantly lower values for all of the dependent variables except heat of decomposition. Yellow fabrics yielded the smallest differences across levels of dye type and dye level. Sharp differences were exhibited by the blue and red fabrics across levels of dye type and dye level, particularly for the peak heights ratio and heat of decomposition.

The MX fabrics, blue fabrics, and 4% fabrics had the earliest temperatures at onset of degradation with the MX Blue 4% fabric exhibiting the lowest value. The MX Blue 4% fabric also yielded the lowest peak heights ratio. The low peak heights ratio for the 4% fabrics indicated more area under the DSC curve was shifted into the second peak for the 4% fabrics and the two peaks were more nearly the same height. Blue fabrics exhibited the greatest differences across the levels of dye type and dye level for the peak heights ratio.

The MX Blue 4% fabric did not yield the lowest values for the temperature at the degradation peaks. Both degradation peak temperatures were lowest for the 4% fabrics and the red fabrics with the H Red 4% fabric having the lowest temperature at both peaks.

For heat of decomposition the significant differences due to color were tied to type and level. Heat of decomposition values for yellow fabrics remained relatively constant across type and level.

Red fabrics yielded an increase across dye type (H to MX) and a decrease across dye level (1% to 4%) while the blue fabrics yielded a decrease across dye type and an increase across dye level.

II. COMPARISON OF DYED AND UNDYED FABRICS

Burning Behavior on the MAFT

All 13 fabrics followed a similar pattern of burning on the MAFT. Following ignition, at a position 4 inches from the bottom edge of the cylindrical fabric specimen, the flames spread upward reaching the top of the MAFT cylinder. The flames spread more slowly toward the bottom edge and generally began moving around the sides of the specimen before the front bottom edge was completely consumed. The last stage of burning was the flame spread at the specimen area directly opposite the specimen ignition location. In some instances the flame spread along the top of the cylinder during the final stage of burning resulted in the unburned back portion of the specimen falling to the floor of the MAFT.

The behavior of the fabrics during this burning pattern was not the same for all 13 fabrics. The undyed fabrics generally hung on the MAFT with very little movement during burning. A few of the undyed specimens drew in slightly toward the rods surrounding the central cylinder and seven of the eight specimens dropped to the floor during the final stage of burning.

The majority of the 1% dyed fabrics drew in slightly toward the MAFT rods but the fabric often returned to its relaxed position as burning continued. In a few cases the fabric specimen drew in slightly and relaxed several times during burning. Over half of the 1% fabric specimens within each fabric group dropped to the floor during the final stage of burning, with the exception of the H Blue 1% fabric. Only two of the eight H Blue 1% fabric specimens dropped.

For the 4% fabric specimens the drawing in behavior was more dramatic. With the exception of the MX Red 4% fabric, all the 4% fabric specimens drew in against the MAFT rods to such an extent that the rods were clearly outlined in the fabric specimen. This drawing in behavior continued throughout the burning of the specimen and also involved a drawing up as well as drawing in.

One fabric specimen of the eight H Red 4% fabrics dropped to the floor during the final stage of burning. None of the other 4% fabric specimens, except the MX Red 4% fabrics, dropped, even though the flame spread across the top of the cylinder as with the 1% fabrics. As the flame spread across the top of the cylinder, the ash of the 4% fabrics was able to support the unburned fabric mass.

The MX Red 4% fabrics behaved more like the 1% fabrics though their drawing in behavior was slightly stronger. Four of the eight MX Red 4% specimens dropped to the floor during the final stage of burning. These findings are consistent with the MAFT heat transfer results for the MX Red 4% fabric. The MX Red 4% fabric exhibited heat transfer values closer to the 1% fabrics than the 4% fabrics.

The importance of these behaviors in relation to some of the MAFT heat transfer variables can also be seen with the other fabrics. The second heat transfer rate peak for the 4% fabrics occurred during the final stage of burning as the back portion of the fabric drew in toward the MAFT cylinder and did not drop to the floor. This behavior contributed to a higher second heat transfer rate peak, a longer time to the second peak, and a higher total heat transfer compared with the 1% fabrics.

Heat Transfer Variables

The mean results for both dyed and undyed fabrics on the MAFT are given in Table II, page 34. Representative heat transfer curves for the dyed fabrics are illustrated in Figures 19-24. A representative undyed heat transfer curve is shown in Figure 25.

The analysis of variance procedure used for the dyed fabrics included a contrast to compare the dyed and undyed fabrics. In addition, a Waller-Duncan Multiple Range post-hoc test was used to identify specific differences between dyed and undyed fabrics. Of the six MAFT dependent variables, only the heat transfer rate at the second peak and total heat transfer were found to have significant differences between dyed and undyed fabrics. The contrast was non-significant for the heat transfer rate at the first peak, the time to reach each of the two heat transfer rate peaks, and total burn time (Tables III-VIII, pages 35-37).

The heat transfer rate at the second peak was lower for the undyed fabric. The Waller-Duncan post-hoc results for this variable indicate the undyed fabric is significantly lower than all the 4% dyed fabrics except the MX Red 4% fabric. Results also indicate the undyed fabric did not have a heat transfer rate at the second peak significantly lower than the 1% fabrics. These findings support those of Hopkins and Buchanan (24) who found that reactive dyed fabrics at dye

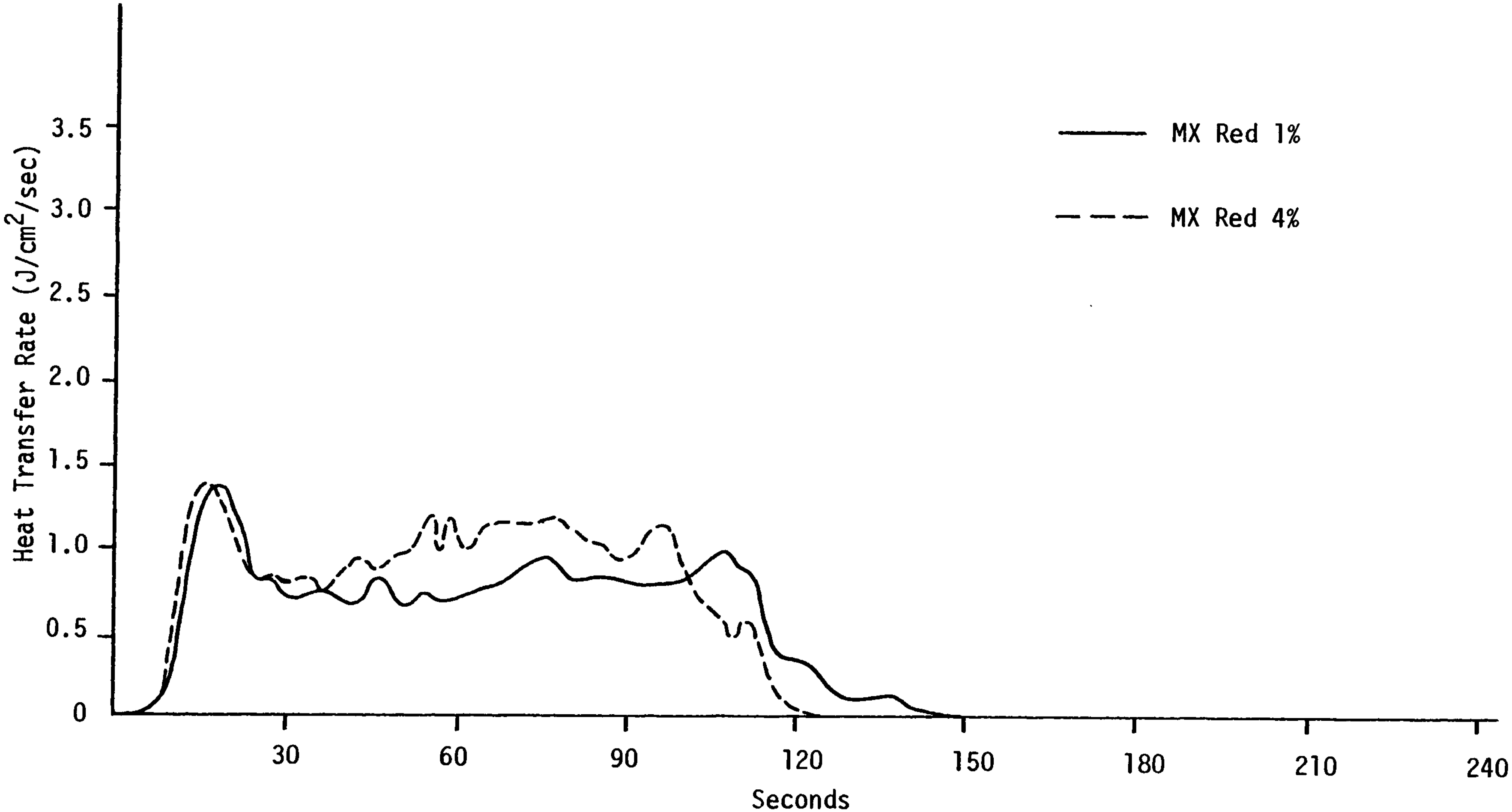


Figure 19. Representative Heat Transfer Curves for Procion MX Red Fabrics.

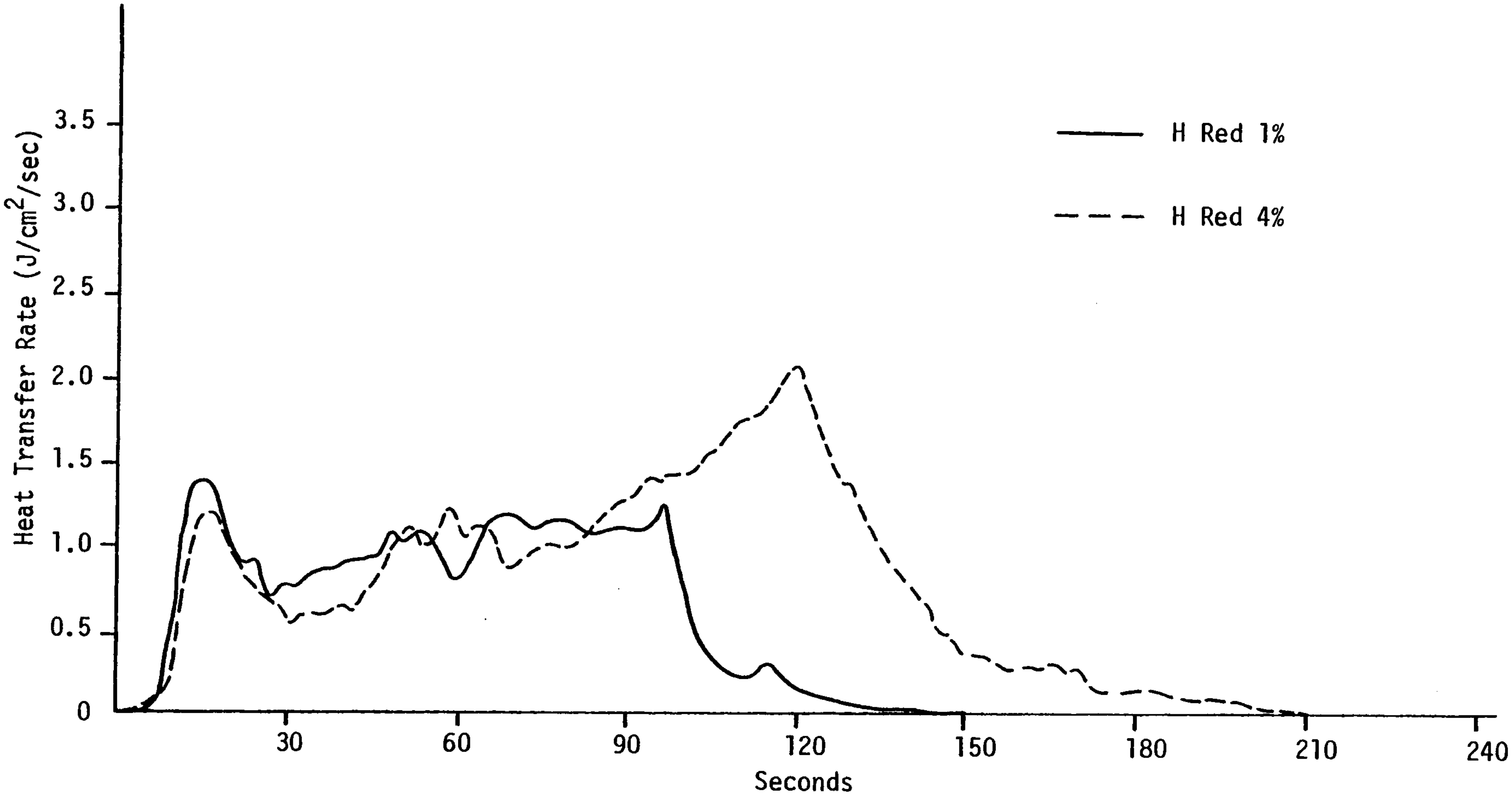


Figure 20. Representative Heat Transfer Curves for Procion H Red Fabrics.

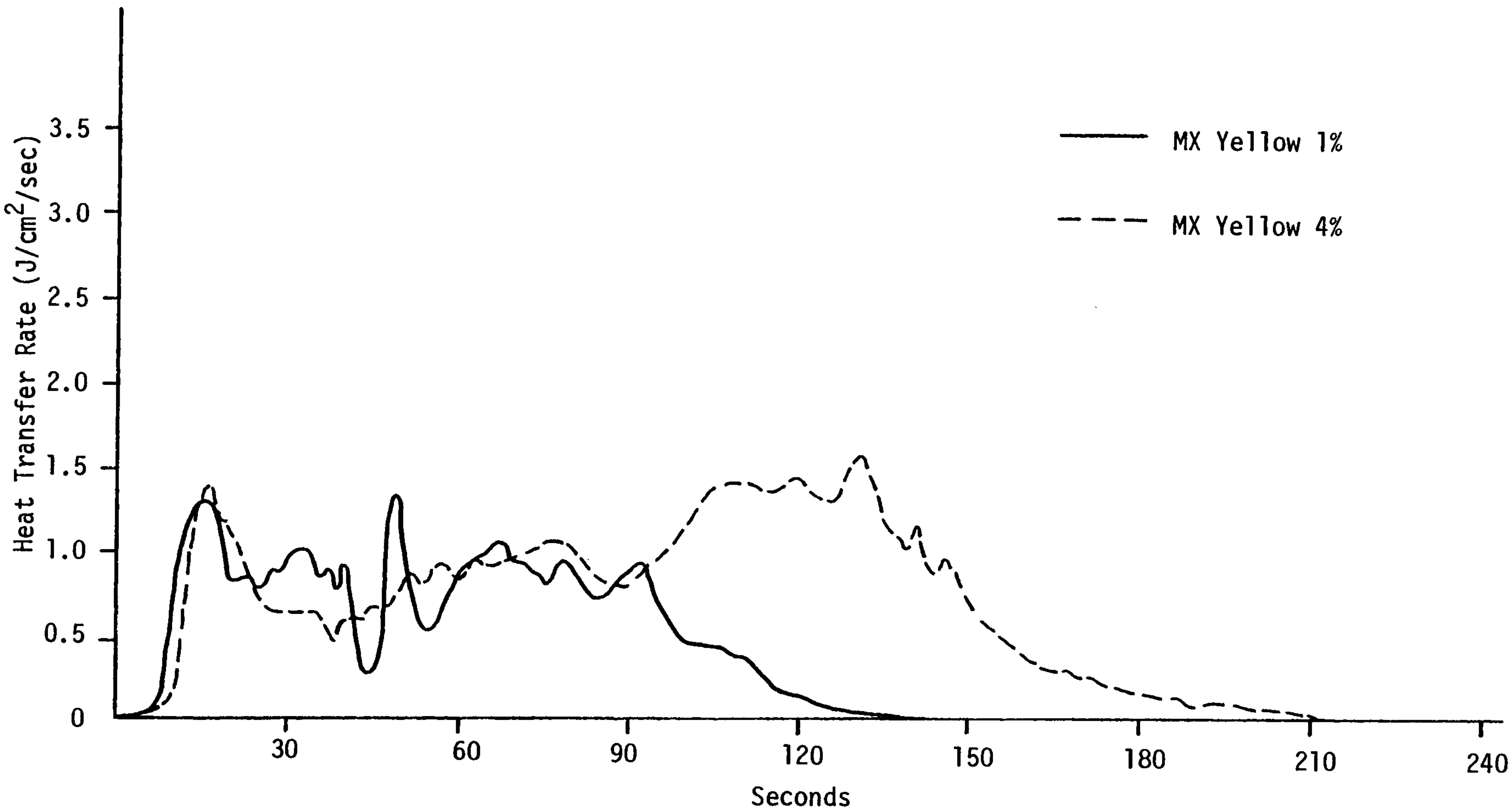


Figure 21. Representative Heat Transfer Curves for Procion MX Yellow Fabrics.

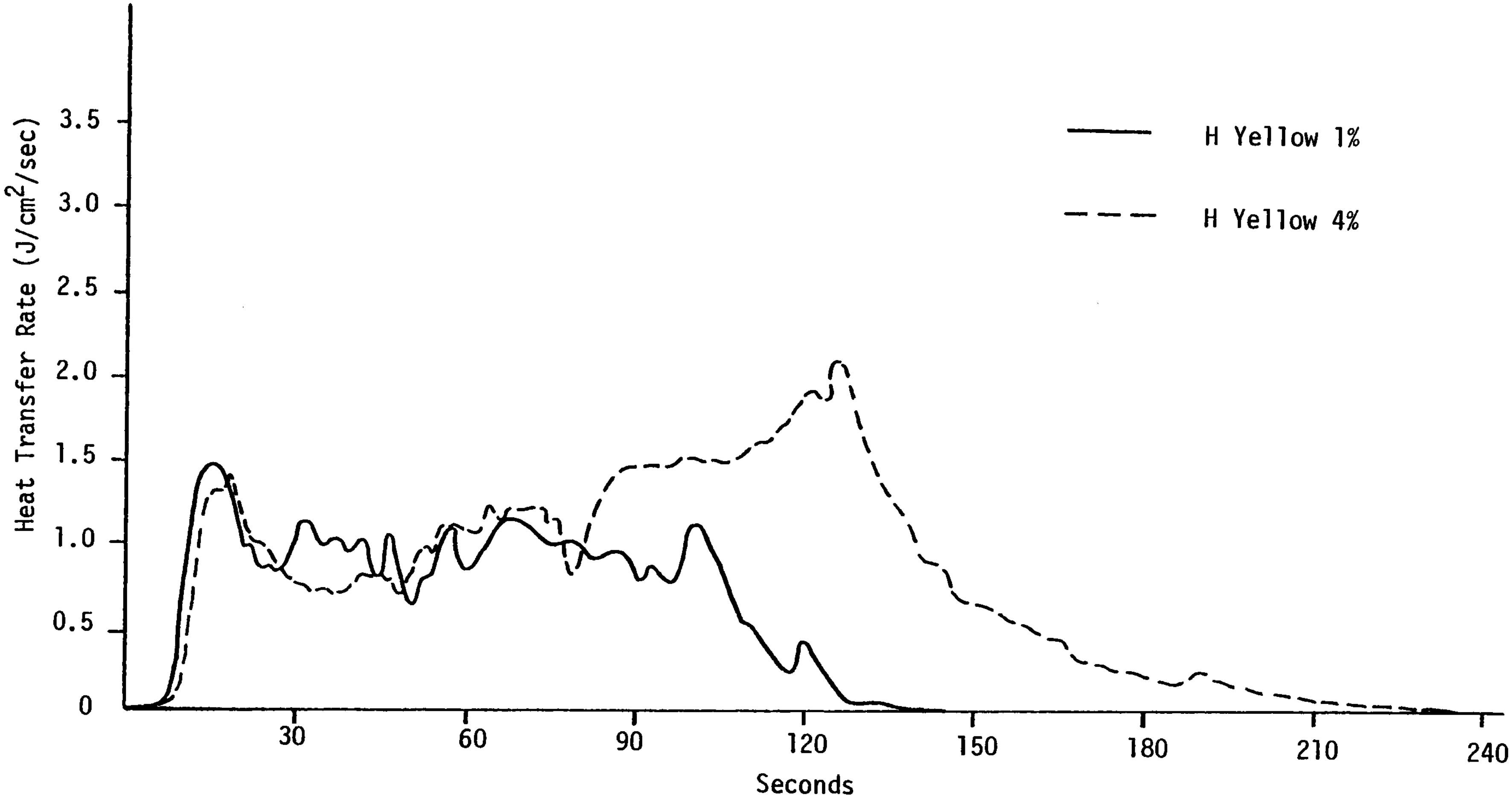


Figure 22. Representative Heat Transfer Curves for Procion H Yellow Fabrics.

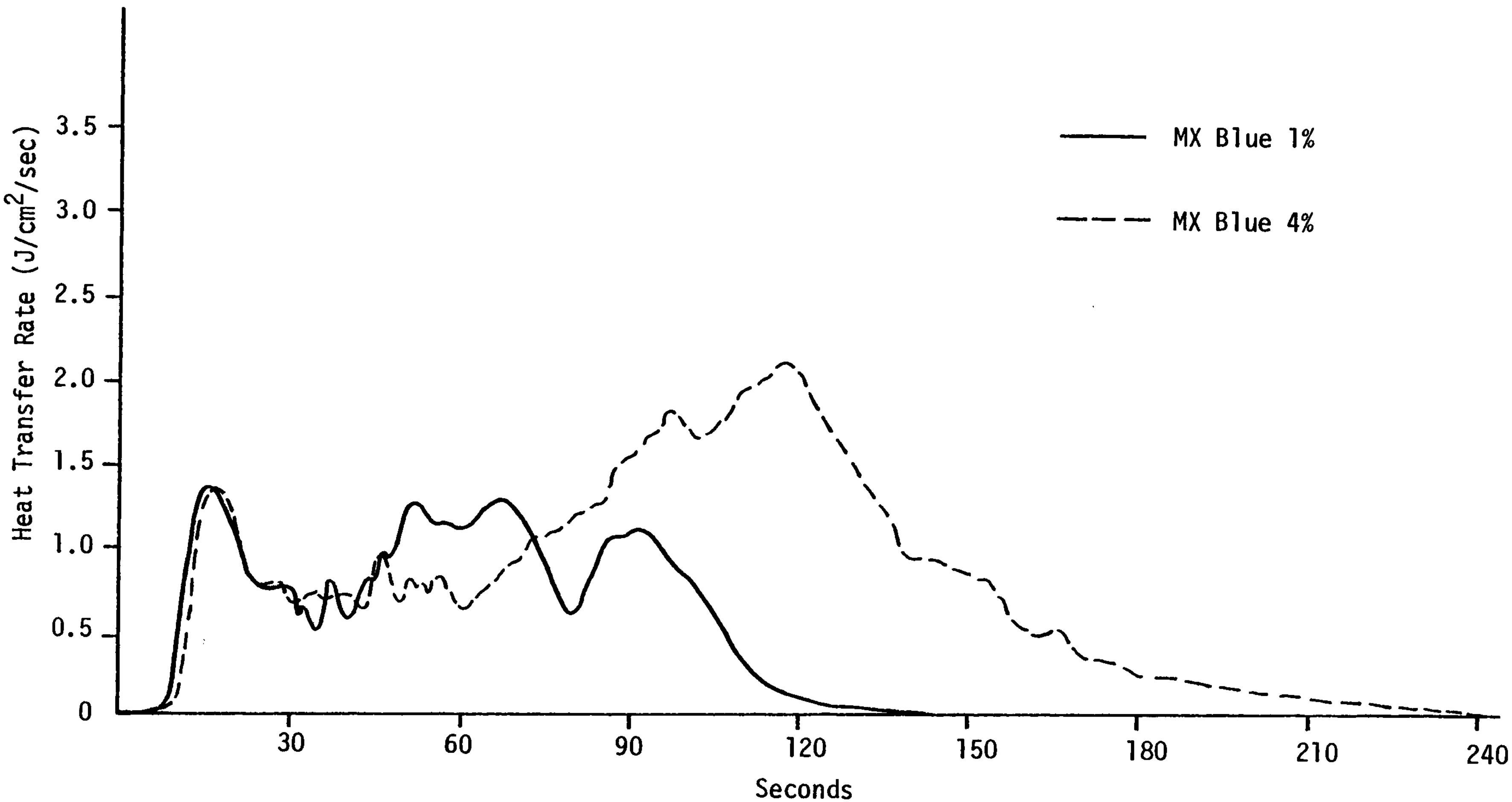


Figure 23. Representative Heat Transfer Curves for Procion MX Blue Fabrics.

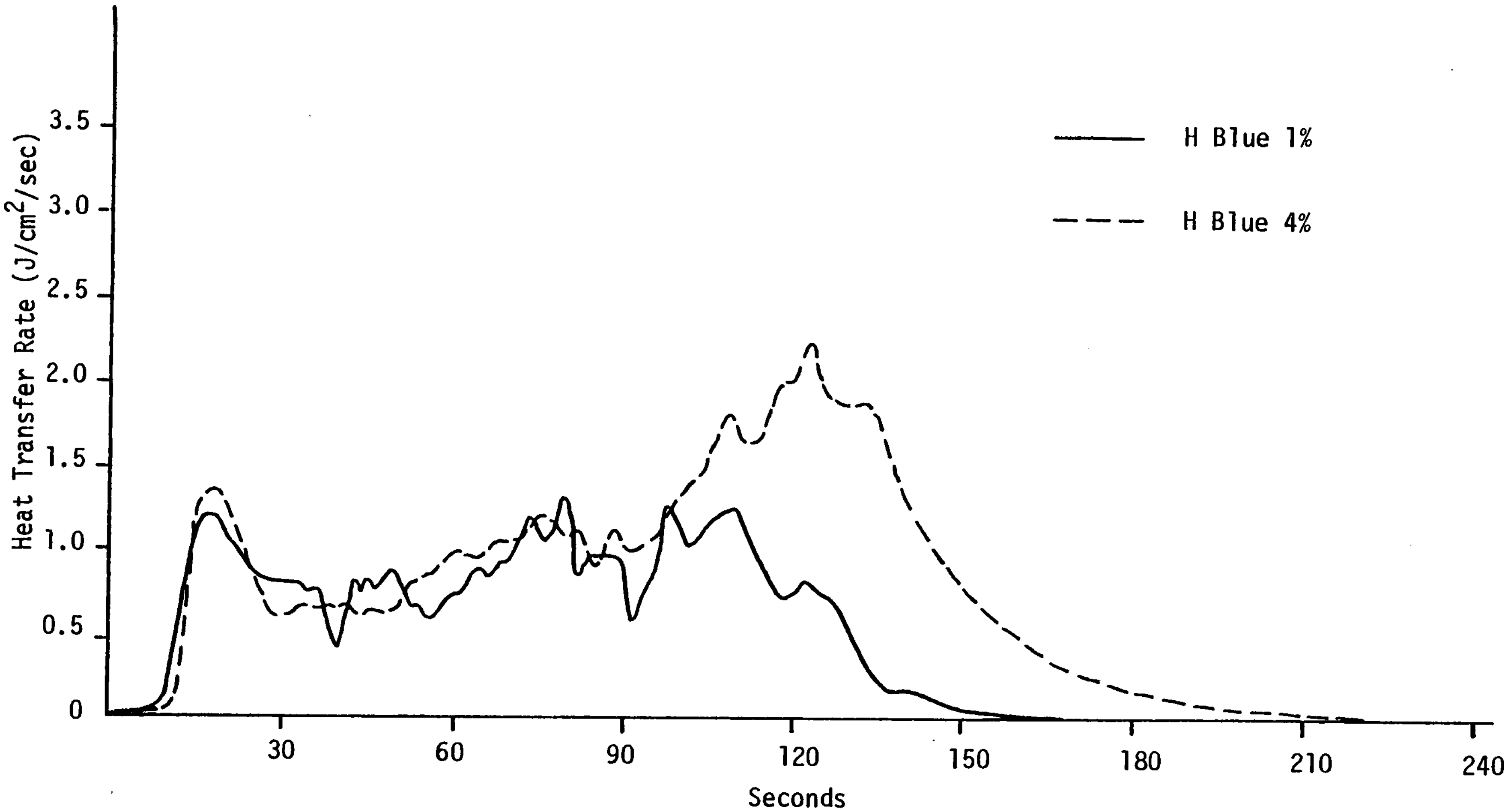


Figure 24. Representative Heat Transfer Curves for Procion H Blue Fabrics.

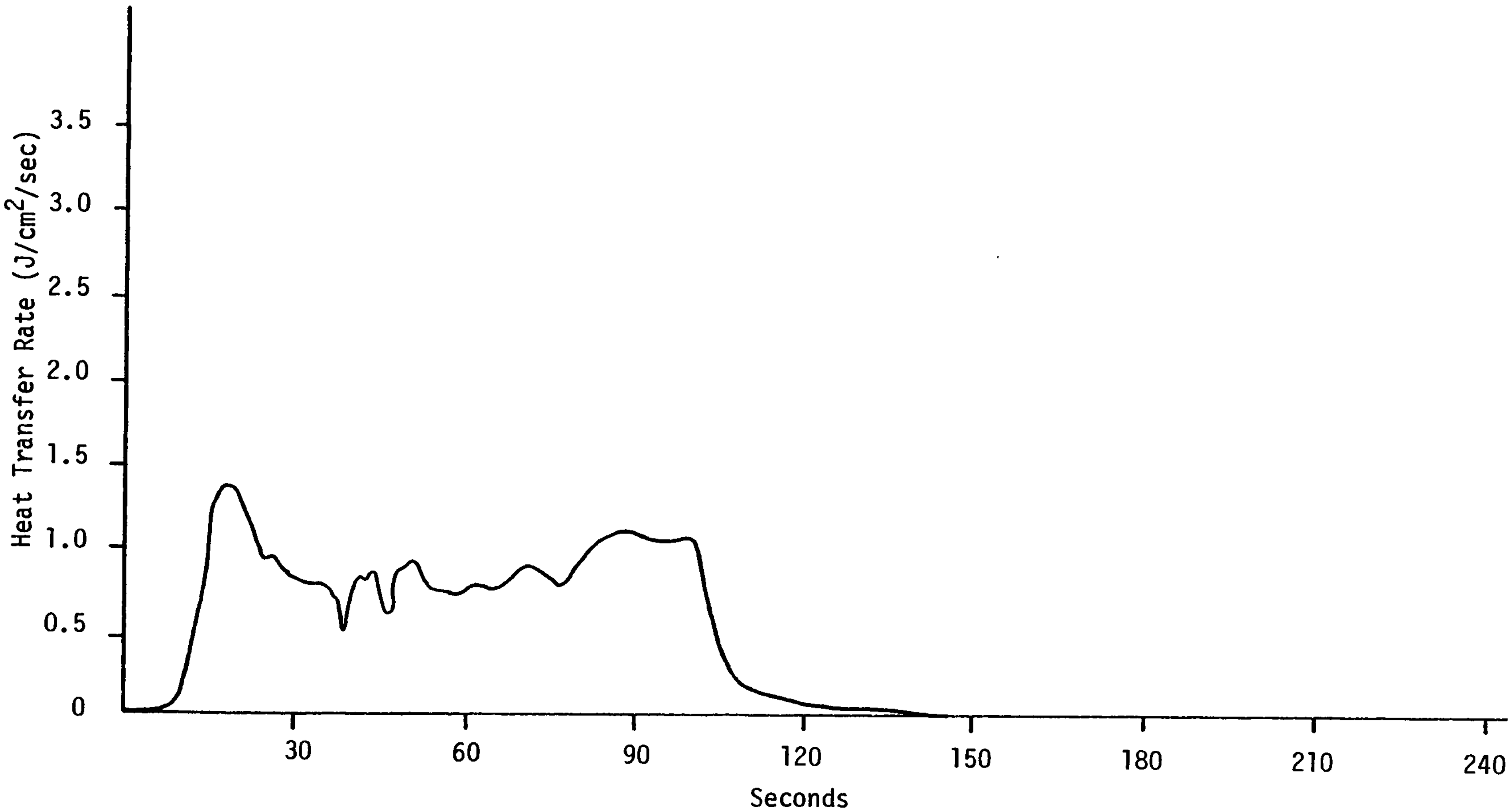


Figure 25. Representative Heat Transfer Curve for Undyed Fabric.

levels less than 2% did not significantly alter the maximum heat transfer rate.

Waller-Duncan post-hoc results for the time to reach the second heat transfer rate peak and total heat transfer were similar to the heat transfer rate at the second peak. The undyed fabric took less time to reach the second peak than the 4% dyed fabrics, except the MX Red 4% fabric, but were not significantly different from the 1% fabrics. Total heat transfer values were significantly higher for all the 4% fabrics, except the MX Red 4% fabric; however, the 1% dyed fabrics were not significantly different from the undyed fabric.

Thus, the fabrics dyed at the 1% level did not exhibit significant differences compared with the undyed fabric for the heat transfer variables. Fabrics dyed at the 4% level had a higher heat transfer rate at the second peak, took longer to reach the second heat transfer rate peak, and transferred more total heat.

Heat/Time Relationship

The calculated heat/time relationship curve for the undyed fabric using the exponential growth curve

$$HE = A(1-e^{-Kt})^{\frac{1}{1-M}}$$

is illustrated in Figure 26. Calculated curves for the dyed fabrics were presented in Figures 7-12, pages 50-55. The A, K, and M values, R^2 values for the curves, and the times to intersect the Derksen injury curve for both dyed and undyed fabrics are listed in Table IX, page 44.

The dyed vs. undyed contrast used in the analysis of variance procedure was found to be significant for parameters A and M

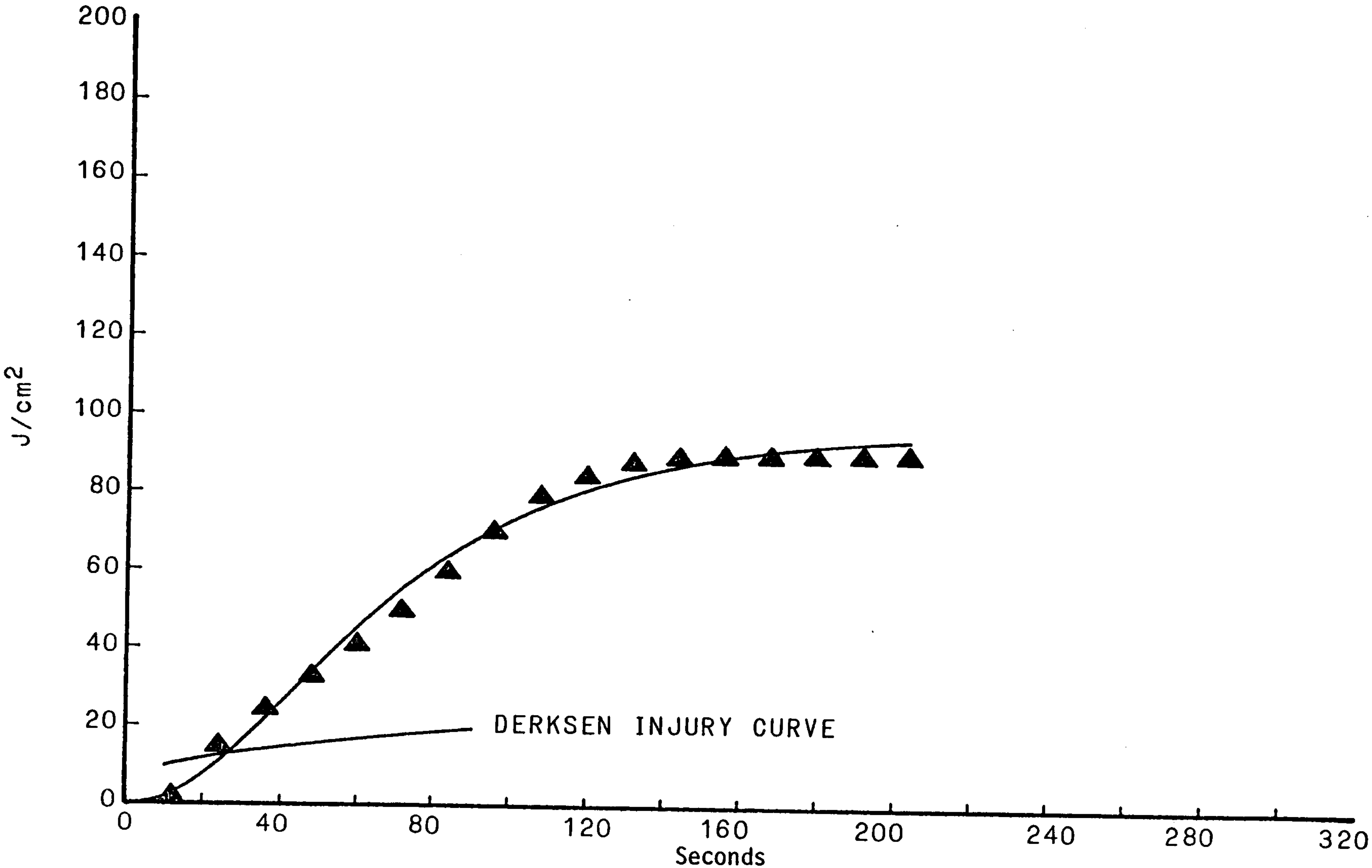


Figure 26. Heat/Time Relationship for Undyed Fabric.

(Tables X-XII, pages 45 and 46). Waller-Duncan post-hoc results indicated that the value for parameter A was significantly lower for the undyed fabric than all the 4% fabrics except MX Red 4%. The undyed and 1% dyed fabrics were not significantly different for parameter A.

Parameter M was also lower for the undyed fabrics. Waller-Duncan post-hoc results showed the undyed fabric was significantly different from all the dyed fabrics except the H Yellow 1% and MX Yellow 1% fabrics. Even though the 4% fabrics have heat/time curves with a fit problem in the area controlled by the M parameter, the data for the 4% fabrics show heat/time values slightly lower than the 1% fabrics of corresponding type and color. Thus, the initial rate of the heat/time curve for the undyed fabric appears to be significantly faster than at least the red and blue fabrics of both dye types and levels. However, the time to intersect the Derksen injury curve for both the calculated heat/time curves and the average heat/time data (Table IX, page 44) did not show a significant difference between dyed and undyed fabrics (Tables XIII and XIV, page 57). Consequently, there does not appear to be a difference in the hazard potential of the dyed and undyed fabrics during the first minute of burning.

Flame Spread

The dyed vs. undyed contrast in the analysis of variance procedure was non-significant for the rate of flame spread. Waller-Duncan post-hoc results indicated the undyed fabric was not significantly different from any of the dyed fabrics except the MX Blue 1% and MX Yellow 1% fabrics.

Since the MAFT heat/time relationship analysis indicated the undyed fabric transferred heat more quickly initially than the dyed fabrics, the undyed fabrics might be expected to have shorter flame spread times than the dyed fabrics. However, the times to intersect the Derksen injury curve indicated no difference between dyed and undyed fabrics and the heat/time data during the first 12 seconds indicated the undyed fabric values are close to the values for the dyed fabrics. Thus, the rate of flame spread results appear to be consistent with the MAFT results.

Degradation Temperature Values

Significant differences between dyed and undyed fabrics were found for the temperature at onset of degradation, the degradation temperature at the first peak, and heat of decomposition (Tables XVIII, XIX, and XXII, pages 62 and 64). The undyed fabric did not exhibit a second degradation peak; therefore, the undyed fabric was not included in the analysis of variance for the degradation temperature at the second peak and the peak heights ratio. Representative DSC curves for the undyed fabric and one of the dyed fabrics are shown in Figure 27.

The onset of degradation occurred at a significantly higher temperature for the undyed fabric. The Waller-Duncan post-hoc test showed the undyed fabric to be significantly different from each of the dyed fabrics. These findings are in agreement with those of Segal et al. (50) who found that cellulose dyed with reactive dyes exhibited an earlier onset of decomposition.

The undyed fabric also exhibited a degradation temperature at the first peak which was higher than the dyed fabrics. Waller-Duncan

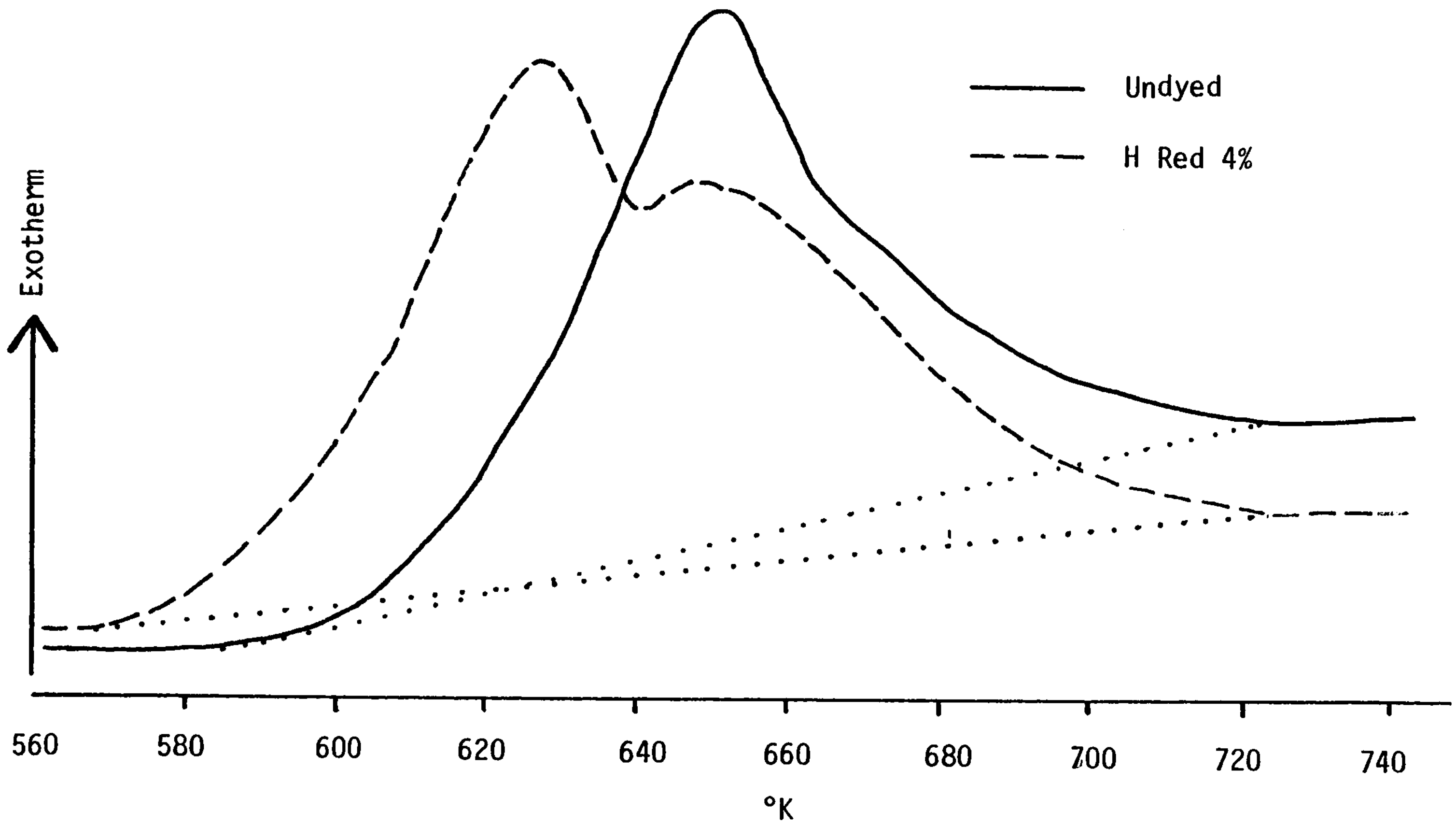


Figure 27. Representative DSC Curves for Dyed and Undyed Fabrics.

post-hoc results revealed that the undyed fabric was significantly higher than each of the dyed fabrics with the exception of the H Blue 1% fabric.

The degradation temperature at the second peak revealed an important difference between the dyed and undyed fabrics. DSC curves for the undyed fabric showed only one peak. This finding suggests that the second degradation peak for the dyed fabrics involves the decomposition of the portions of the cellulose molecules containing dye molecules.

The significant difference between dyed and undyed fabrics for heat of decomposition showed the undyed fabric required less energy for decomposition. According to the Waller-Duncan post-hoc test the undyed fabric had a significantly lower heat of decomposition than each of the dyed fabrics except the H Blue 1% and MX Blue 4% fabrics.

CHAPTER V

SUMMARY, CONCLUSICNS, AND RECOMMENDATIONS

The purpose of this study was to investigate the flammability behavior of reactive dyed cotton fabrics and to compare these results to the flammability behavior of undyed cotton fabric. A 2 x 3 x 2 factorial design was used to evaluate the effects of dye type, dye color, and dye level on the peak heat transfer rates, time to reach the peak heat transfer rates, total burn time, total heat transfer, heat/time relationship, rate of flame spread, and heat of decomposition of the dyed fabrics. The reactive dyed cotton fabrics included two dye types (Procion H and MX), three dye colors (red, yellow, and blue), and two dye levels (1% and 4%). Testing was carried out on the MAFT, the 45° angle tester, and the DSC. In addition, undyed fabric was tested for comparison with the dyed fabrics.

I. HEAT TRANSFER

The 13 fabrics exhibited no difference in heat transfer rate at the first peak, the time to reach the first heat transfer rate peak, and total burn time as measured by the MAFT. Dye type significantly affected the time to reach the second heat transfer rate peak and total heat transfer with the Procion H fabrics exhibiting the highest values. Total heat transfer was also affected by dye color with the blue fabrics yielding the highest values.

The effect of dye level was observed as an increase in the values for the heat transfer rate at the second peak, the time to reach the second heat transfer rate peak, and total heat transfer. The MX Red 4% fabric proved to be the exception for the 4% fabrics. This fabric produced values similar to the 1% fabrics. Further analysis of this fabric, beyond the visual examination and reflectance readings carried out in this study, is needed to provide an understanding of this fabric's behavior.

Dye level was also important in the differences between dyed and undyed fabrics. The undyed fabric produced lower values than the 4% dyed fabrics for the heat transfer rate at the second peak and total heat transfer. Undyed fabric values for these variables were similar to the 1% fabrics.

Heat/time data analysis provided valuable information about the flammability hazard of the 13 fabrics tested. The heat/time data for the 13 fabrics showed a slow initial rate of heat transfer followed by a rapid acceleration in the rate and finally a leveling off as the total amount of heat transferred by the fabric was reached. The amount of heat transferred by the 4% fabrics did not surpass the 1% and undyed fabrics until after 60 to 100 seconds of burning. The times for the average heat/time data to cross the Derksen injury curve indicated these fabrics become hazardous after approximately 21 seconds of burning. No difference was found between the dyed fabrics or between the dyed and undyed fabrics in the time for the average heat/time data and the Derksen injury curve to intersect. Consequently, there is no difference in the flammability hazard of these fabrics during the first minute of burning.

The regression analysis used to establish representative heat/time curves for these fabrics provided interesting results. The parameter values showed that parameter A corresponds well with total heat transfer. Parameter K decreased and parameter M increased as dye level increased. Parameter M was also significantly lower for the undyed fabrics over the dyed fabrics. The parameter values indicated the 4% fabrics transferred heat more slowly initially than the 1% fabrics but ultimately transferred more heat. The times for the calculated curves to cross the Derksen injury curve also exhibited this difference due to dye level, with the 4% fabrics taking longer to cross the Derksen injury curve.

Visual analysis of the calculated heat/time curves and heat/time data and a comparison of the Derksen injury curve intersection times for the calculated heat/time curves and the heat/time data indicated the calculated heat/time curves did not fit the data in the first 50 to 80 seconds well enough to provide accurate results, particularly for the 4% fabrics. Further work is needed to establish calculated heat/time curves which provide an accurate picture of the heat/time relationship for fabrics burned on the MAFT. Since the derivative of a curve established by the equation

$$HE = A(1-e^{-Kt})^{\frac{1}{1-M}}$$

is a one peaked curve and these fabrics yield heat transfer rate curves with two peaks, heat/time curves for these fabrics might be established more accurately by analyzing the two peak sections of the curve separately.

The burning behavior of the dyed fabrics on the MAFT also suggests further study. The shrinkage behavior during the burning of the dyed fabrics, which increased with increasing dye level, raises questions about the thermal and physical properties of reactive dyed cellulose.

II. FLAME SPREAD

Only dye level affected the rate of flame spread for the dyed fabrics. As dye level increased flame spread time increased; however, all differences were less than two seconds. Dyed and undyed fabrics exhibited no difference in rate of flame spread.

Flame spread results were consistent with the MAFT results. Findings indicated that the 45° angle test did not provide any information for these fabrics beyond that which can be obtained through a heat/time relationship analysis of MAFT data.

III. DECOMPOSITION

Heat of decomposition results did not show any clear relationship to the results of the heat transfer testing. Undyed fabrics require less energy for decomposition than the dyed fabrics. No clear difference between fabrics dyed at the 1% and 4% level was found; however, differences between colors were tied to type and level. Heat of decomposition values for yellow fabrics remained relatively constant across dye type and dye level while the red and blue fabrics exhibited contrasting increases and decreases across dye type and dye level.

Dye level was significant for the other DSC variables. The effect of level was observed as a decrease in the values for the temperature at onset of degradation, degradation peak temperatures, and the peak heights ratio. Dye color was also significant for all of these variables except the peak heights ratio. Again, yellow fabrics yielded the smallest differences and the red and blue fabrics exhibited sharp differences across dye type and dye level. Blue fabrics exhibited the lowest temperature at the onset of degradation and red fabrics exhibited the lowest temperatures at both degradation peaks.

Results for these variables for the undyed fabric showed that the undyed fabric begins to degrade at a higher temperature, exhibits only one degradation peak and exhibits a higher degradation peak temperature than the dyed fabrics. Thus, the second degradation peak for the dyed fabrics may represent the decomposition of portions of the cellulose molecule containing dye molecules.

The DSC results do not seem to provide any additional flammability hazard information in comparison with the MAFT results. However, the MAFT and DSC results do indicate that the amount of reactive dye in cellulose is the most important factor in heat transfer during burning while reactive dyed cellulose decomposition is closely tied to both the chemical structure of the dye, including the dye chromophore, and the amount of dye.

The results of this study combined with previous findings on the flammability behavior of other types of fabrics dyed with different classes of dyes provide the kind of information needed for apparel flammability to be approached realistically. These results along with previous evaluations on the influence of fiber content, fabric weight, flame retardants, and fabric layers on flammability behavior provide a valuable framework for the design of a flammability hazard index for consumers. Further work involving different combinations of fiber content, fabric weight, fabric construction, and fabric layers which are appropriate in relation to the consumer market could enhance such a flammability hazard index.

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APPENDIX

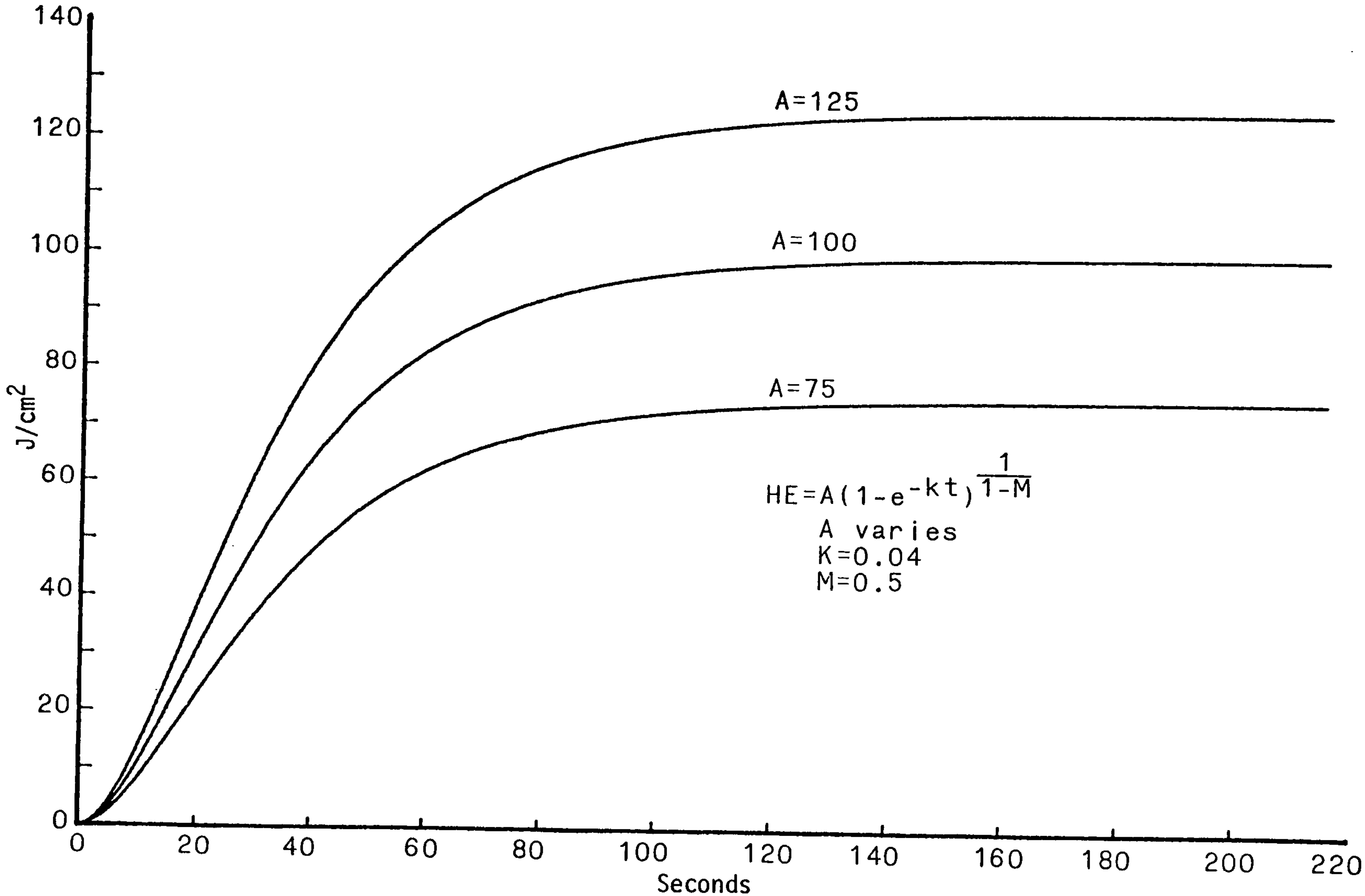

Figure 28. Effect of Parameter A on Calculated Heat/Time Curves.

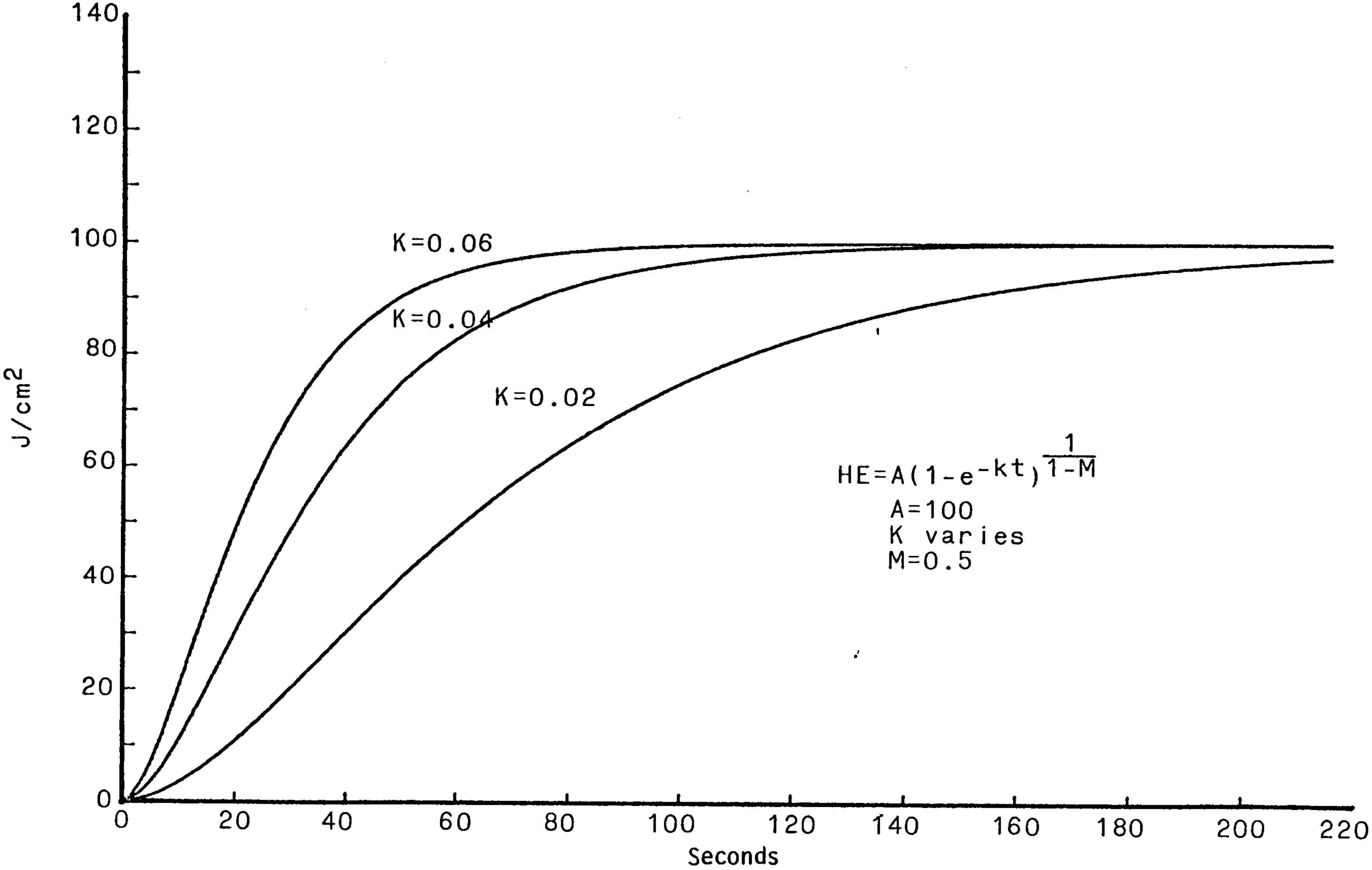

Figure 29. Effect of Parameter K on Calculated Heat/Time Curves.

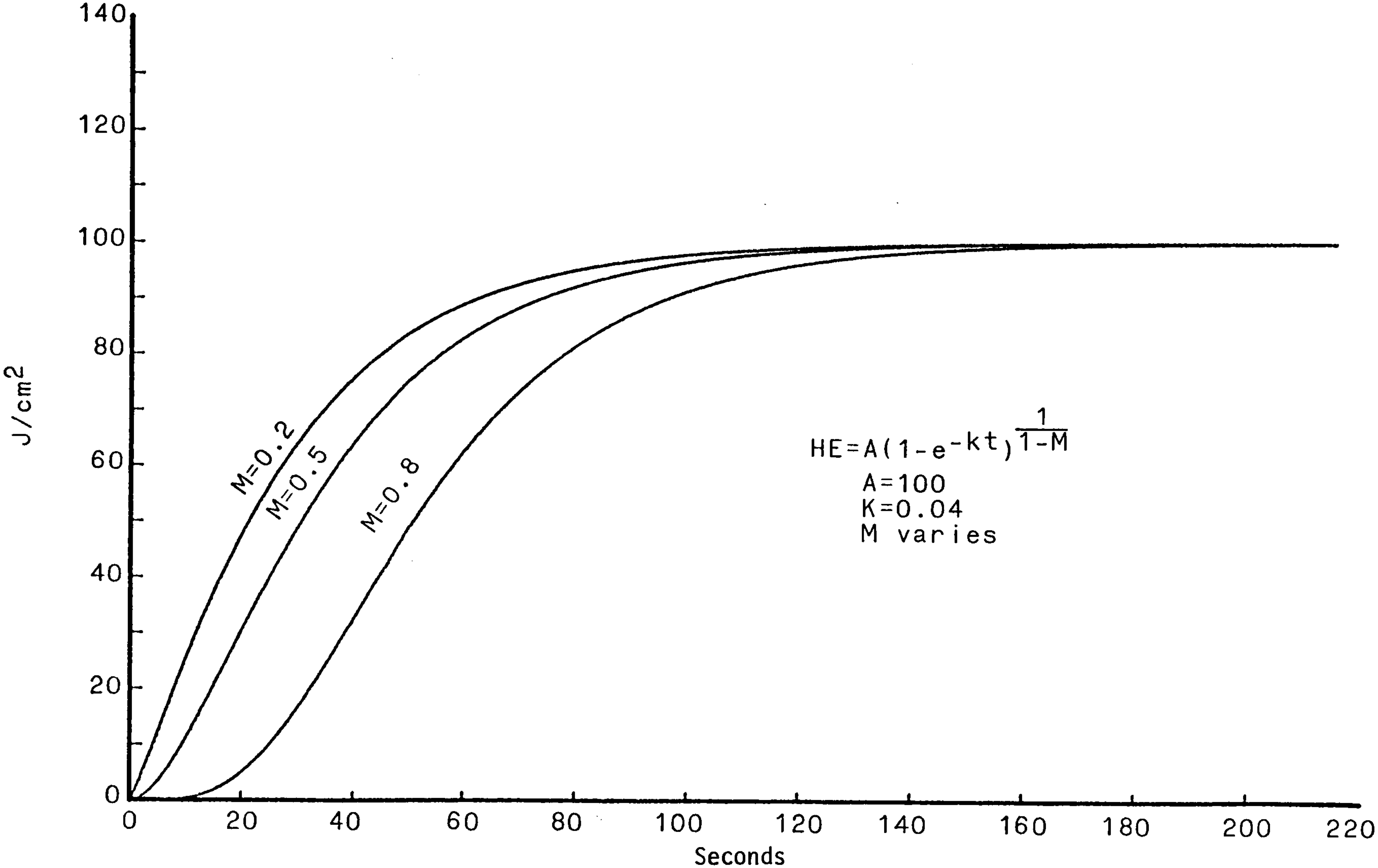

Figure 30. Effect of Parameter M on Calculated Heat/Time Curves.

VITA

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